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**PUBLIC  
DOCUMENT**

January 29, 2003

Mr. John McGuiggin, PE  
U.S. DOT/RSPA/Volpe Center/DTS-33  
55 Broadway, Kendall Square  
Cambridge, Massachusetts 02142

**SDMS Document ID**



**1008489**

**Subject:** Contract DTRS57-99-D-00017, Task Order C0023  
U.S. EPA Region 8, Asbestos Project - Emergency Response  
Libby Sister Site Field Sampling Activities at  
Former Vermiculite Intermountain Facility - SLC2  
333 West 100 South, Salt Lake City, UT  
Summary Report (Revision 1)

Dear Mr. McGuiggin:

CDM Federal Programs Corporation (CDM) is pleased to submit this summary report, with incorporated comments, for the field sampling activities that occurred October 14 through October 16, 2002 at the former Vermiculite Intermountain facility at 333 West 100 South, Salt Lake City, UT (SLC2). The scope of services performed by CDM is pursuant to the scope of work included in the task order/technical direction referenced above. This report presents background information, site description, site history, summary of field activities, and soil and air sampling results that took place at SLC2.

### **Background**

The U.S. Department of Transportation's John A. Volpe National Transportation Systems Center (Volpe Center) has an Interagency Agreement (IAG) with the U.S. Environmental Protection Agency (EPA) Region 8 for environmental engineering and related support. Volpe Center support includes activities such as preparation of technical documents, development of program management plans, performance of environmental assessments/investigations, and assistance on remediation projects including emergency response.

Since November 1999, Volpe Center's Environmental Engineering Division (DTS-33) has been providing EPA Region 8 with immediate environmental engineering and site assessment support at Libby, Montana. As part of this work, the Volpe Center has provided support on investigations to monitor, sample and characterize asbestos-containing materials that may be present in the Libby community and in areas of former vermiculite mining and processing activities. EPA Region 8 has also expanded the investigations outside of the Libby Valley to determine other potentially contaminated properties that may have been impacted as a result of vermiculite processing and handling facilities. In support of these investigations, EPA Region 8 requested the Volpe Center and its contractor CDM to conduct field-sampling activities at areas



where vermiculite and/or amphibole asbestos may have been introduced during operations at SCL2. EPA Region 8 identified this location from U.S. Geological Survey (USGS) and Bureau of Mines publications, as a site that received ore or vermiculite from Libby, Montana.

The overall objectives of the field-sampling activities were to:

- Collect surface and subsurface soil samples in the area where the plant once existed to determine if Libby amphibole (LA) asbestos contamination is present.
- Collect ambient air samples during the sampling effort to determine whether or not there are airborne LA structures are present.

This report summarizes the field activities that took place during the October 14 through October 16, 2002 field investigation.

### **Site Description**

SLC2 is located at 100 South 330 West just south of the Delta Center (Attachment 1, Figure 1). The site is situated adjacent to a power transfer station and an asphalt parking lot. Site detail of SLC2 is illustrated on Figure 2 (Attachment 1). The aerial photograph shown in the figure was taken in 2000 and obtained from Olympus Aerial Surveys, Inc. According to historical records, SLC2 was the original location for the Intermountain Insulation Company (formerly Vermiculite Intermountain) vermiculite processing facility. The former processing facility is now demolished and the site is currently owned in part by Pacific Corporation (PacifiCorp), a parent company of Utah Power and Light. The original plant boundaries probably encompassed adjacent properties including the asphalt parking lot and a storage business.

### **Site History**

The exfoliation facility was formerly known as Vermiculite Intermountain. The company later changed its name to Intermountain Insulation (date unknown). Vermiculite-containing material was shipped to SLC2 via railcars. According to interviews with a previous employee, the material was scattered about the property from both leakage from the rail cars and from the transfer of the material from the railcars to the processing plant.

Historical research conducted by the EPA On-Scene Coordinator prior to the sampling activities indicated that Intermountain Insulation had operated at this site from about 1940 to 1984 before relocating their operations to another site at 733 West 800 South (SLC1). Intermountain Insulation, under license to W.R. Grace Construction Products Division, manufactured and distributed insulation, fireproofing, vermiculite soil conditioner, masonry fill and concrete plaster aggregate until the company went bankrupt in 1987.

### **Summary of Field Activities**

The field activities were conducted on October 14 through October 16, 2002 by Frank Morris (CDM) and Melissa Petrak (PES). Prior to field activities, a signed access agreement from



PacifiCorp was obtained by the EPA. In addition, a utilities meet was conducted through the Utah Utilities Location Center (e.g. Blue Stakes, Utah One Call) to identify underground utility locations so they would not be disturbed during field activities. CDM received confirmation that all proposed sampling locations were clear of underground utility paths. However, because of the proximity of the site to the adjacent substation, a representative from PacifiCorp was onsite during the soil sampling event to ensure underground utilities associated with the substation were not disturbed.

Prior to performing any field activities, all field team members reviewed the site-specific health and safety plan. Field team members collecting soil samples were outfitted in level C personal protective equipment (PPE).

A total of 100 soil samples were collected from the SLC2 site. Specifically, soil samples were collected within a fenced in gravel lot directly east of the substation where the former processing facility once existed (Attachment 1, Figure 2). Soil samples were not collected from the area where the rail spur originally existed due to high voltage lines beneath the surface. One personal air and one ambient air sample was collected during the sampling effort. Four clearance air samples were collected the day after the sampling effort. Field sample data sheets (FSDS) were completed for all samples collected and are included in Attachment 2. Digital photographs of the site were recorded during the sampling effort and are included in Attachment 3.

### **Soil Sampling**

The fenced in gravel lot east of the substation was segregated into 32 grids measuring approximately 25 by 25 feet (ft). Soil sample locations were positioned at the node of each grid. A discrete grab sample was collected at 0 to 2, 2 to 6, and 6 to 12-inch depth intervals from each sample location. In addition to the grid samples, three individual surface soil samples were collected from areas where gross visible vermiculite was observed. These areas included the lower level and upper level of the exposed building foundation of the former processing facility and the former railroad spur location (Attachment 1, Figure 3).

A tractor mounted direct-push technology (DPT) sampler was used to collect the soil samples. Once a soil core was extracted, depth intervals were measured for sampling purposes. All soil samples were collected using disposable trowels and placed into zip lock baggies. DPT equipment was decontaminated between samples using deionized water and Alconox soap. All soil samples were collected in accordance with the sampling and analysis plan for Libby Sister Sites (CDM 2001). Soil samples were sent under chain of custody to the CDM laboratory in Denver, Colorado for further processing (i.e., drying, splitting, etc.). Once the soil samples were processed, they were submitted for LA analysis by polarized light microscopy (PLM) (NIOSH 1994a). Table 1 in Attachment 4 indicates the index ID, grid location, sample depth, analytical result, and product observation for each soil sample collected.

While vermiculite was observed in only four surface soil samples collected, analytical results indicated that at least trace amounts (less than or equal to  $\leq$  1 percent [%]) of LA

# CDM

contamination, extended throughout the entire fenced in area (Attachment 1, Figure 3). From the 35 surface soil samples collected, 30 samples had  $\leq 1\%$  LA contamination while three samples had 2 to 3% LA. The surface soil sample with the highest LA concentration was collected from grid G34 with 7% LA. Only one sample collected was nondetect (G32).

LA contamination extended beyond the surface (2 inches) layer (Attachment 1, Figure 4). Thirty-two mid-range (2 to 6 inches) subsurface soil samples were collected. Twenty-seven samples had  $\leq 1\%$  LA contamination. Grids G04, G05, and G06 had 12%, 4%, and 2% LA, respectively. Twelve samples collected from this range had some type of visible vermiculite within the soil matrix. Only two samples were nondetect (G00 and G34).

Beyond 6 inches, fewer samples had detectable levels of LA asbestos (Attachment 1, Figure 5). A total of 32 subsurface (6 to 12 inches) soil samples were collected. Twenty-two samples had  $\leq 1\%$  LA contamination. Elevated LA contamination was found in samples collected from grids G00/G05 (2%), G03 (3%), G15 (12%), and G04 (15%). In grid G04, visible vermiculite was abundant throughout the entire core. In efforts to determine the lower extent of contamination in grid G04, the DPT core was extended past the lower sampling depth of 12 inches. Refusal was observed at 9.5 ft with visible vermiculite throughout the entire core. An individual soil sample was collected from the 36 to 42 inch depth interval and had the highest LA concentration of all soil samples collected at 18%. Five subsurface soil samples were nondetect.

## Air Sampling

Air samples were collected during the sampling effort to determine LA exposure levels during sampling and whether or not airborne LA structures were present at the site. Due to time constraints, background air samples were not collected as directed by Joyce Ackerman (EPA) and Paul Kudaruskas (Volpe Center).

A personal and stationary air sample was collected during the one-day sampling effort. The personal air sample was collected from the breathing zone (BZ) of Frank Morris (sampler) and the stationary air sample was collected from the surface of the toolbox (DPT tractor). Both samples were submitted for LA asbestos by transmission electron microscopy (TEM) using Asbestos Hazard Emergency Response Act (AHERA) counting methods (NIOSH 1994b). Four LA structures were detected on the personal air sample while one LA structure was detected on the stationary air sample. Calculated sample results are presented in Attachment 4, Table 2.

Clearance samples were collected the following day to determine if any residual contamination remained in the air after the sampling effort (Attachment 1, Figure 2). Four stationary air samples (S005 through S008) were collected around the perimeter of the fenced in gravel lot. All four clearance samples were submitted for TEM/AHERA and were nondetect. Calculated sample results are presented in Attachment 4, Table 2.

## Conclusions

- Thirty out of 35 surface (0 to 6 inch) soil samples collected from the fenced in gravel lot had





≤ 1% LA contamination. Four surface soil samples had greater than (>) 1% LA contamination with the highest value being 7%. Only one surface soil sample was nondetect. Visible vermiculite was observed in four surface samples.

- Twenty-seven out of 32 mid-range (2 to 6 inches) subsurface soil samples had ≤ 1% LA contamination. Three samples had > 1% LA contamination with the highest value being 12%. Visible vermiculite was observed in 12 mid-range samples.
- Twenty-two out of 32 subsurface (6 to 12 inches) soil samples had ≤ 1% LA contamination. Five samples had LA contamination > 1% with the highest value being 15%. An additional subsurface soil sample was collected from grid G04 at a depth of 36 to 42 inches had 18% LA contamination. Visible vermiculite was observed in 12 subsurface samples.
- LA structures were detected on the personal air sample (4 structures) and the stationary air sample (1 structure) during the sampling effort indicating some structures were released during sampling. Clearance samples collected the following day were nondetect indicating there was no residual contamination in the air after the sampling effort.

#### Attachments

A site location map is presented in Attachment 1 (Figure 1). A site detail map and soil sample analytical results per depth are presented in Figures 2 through 4 (Attachment 1). Clearance sample locations (Figure 2) are also included in Attachment 1. Completed FSDS are included in Attachment 2. Digital photographs recorded during the sampling effort are included in Attachment 3. Attachment 4 contains analytical results for the soil samples (Table 1) and air samples (Table 2) collected.

If you have any questions, please call me at (617) 452-6257.

Sincerely,

CDM FEDERAL PROGRAMS CORPORATION

for Timothy B. Wall  
Associate and Project Manager

#### Attachments

cc: Paul Kudarauskas (Volpe Center)  
Joyce Ackerman (EPA Region 8)  
Frank Morris (CDM Denver)



**References:**

CDM. 2001. Revision 1 Sampling and Analysis Plan for Libby Sister Sites (Asbestos Project) - Emergency Response and Preliminary Assessment Support, EPA Region 9. February.

NIOSH. 1994a. Asbestos (bulk) by PLM, Method 9002. Issue 2. August.

NIOSH. 1994b. Asbestos by TEM, Method 7402. Issue 2. August.

**Attachment 1**

**Figure Maps of SCL 2**

Figure 1. Site location map of SLC2



**Attachment 2**

**Field Sample Data Sheets**

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
Address: 333 W. 1st 100 South Owner: Utah Power (Pacific Corp.)  
Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3001</u>	<u>IR8-3002</u>	<u>IR8-3003</u>
Location ID	<u>G-00</u>	<u>G-00</u>	<u>G-00</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> FD	<u>FS</u> FD	<u>FS</u> FD
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples	<u>Grab</u> Comp. # subsamples	<u>Grab</u> Comp. # subsamples
Sample Time	<u>0920</u>	<u>0921</u>	<u>0922</u>
Top Depth (in.) *	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.) *	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments 23 22 21 20 19 18 17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 10 20 30 40	<u>* Below coarse gravel</u>	<u>trace of product</u>	<u>→</u>
Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

**LIBBY SISTER SITES FIELD SAMPLE DATA SHEET**  
**SOIL-LIKE MATERIALS**

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
Sampling Team: (circle) CDM PES Other: \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3004</u>	<u>IR8-3005</u>	<u>IR8-3006</u>
Location ID	<u>TEC 121102 G-02 G-01</u>	<u>TEC 121102 G-02 G-01</u>	<u>TEC 121102 G-02 G-01</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> FD	<u>FS</u> FD	<u>FS</u> FD
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>0930</u>	<u>0931</u>	<u>0932</u>
Top Depth (in.) *	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.) *	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>Trace ?</u> →		
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other Industrial  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3007</u>	<u>IR8-3008</u>	<u>IR8-3009</u>
Location ID	<u>TE-03 G-02</u>	<u>TE-03 G-02</u>	<u>TE-03 G-02</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	<u>ES</u> FD	<u>ES</u> FD	<u>ES</u> FD
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>0940</u>	<u>0941</u>	<u>0942</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>Product Trace</u> <u>1' refusal</u> <u>Concrete Foundation</u>	<u>Visible</u>	
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____



**LIBBY SISTER SITES FIELD SAMPLE DATA SHEET**  
**SOIL-LIKE MATERIALS**

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
Land Use: (circle) Residential School Commercial Mining Roadway Other Industrial  
Sampling Team: (circle) CDM PES Other: \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3010</u>	<u>IR8-3011</u>	<u>IR8-3012</u>
Location ID	<u>REC 121402 G-04 G03</u>	<u>REC 121402 G-04 G03</u>	<u>REC 121402 G-04 G03</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u> Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle) <u>FS</u> FD	<u>FS</u> FD	<u>FS</u> FD	<u>FS</u> FD
Matrix Type (circle) <u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle) <u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>0950</u>	<u>0951</u>	<u>0952</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments			<u>Abundant Product 3-4'</u>
Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other Industrial  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3013</u>	<u>1R8-3014</u>	<u>1R8-3015</u>
Location ID	<u>121 G-05 G04</u>	<u>121 G-05 G04</u>	<u>121 G-05 G04</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	<u>FS</u> FD _____	<u>FS</u> FD _____	<u>FS</u> FD _____
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1000</u>	<u>1001</u>	<u>1002</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>Some brown soil fill first 6"</u>	<u>Abundant Product 6"</u>	<u>4'</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

Redrill offset for visual estimate of depth refusal @ 9.5' product full length

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other Industrial  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3016</u>	<u>1R8-3017</u>	<u>1R8-3018</u>
Location ID	<u>121205 606 605</u>	<u>121205 606 605</u>	<u>121205 606 605</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1010</u>	<u>1011</u>	<u>1012</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>tan soil fill</u> <u>trace of product</u>	<u>Abundant</u> <u>Product</u>	<u>Brick @</u> <u>1'</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3019</u>	<u>IR8-3020</u>	<u>IR8-3021</u>
Location ID	<u>606</u>	<u>606</u>	<u>606</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>ES</u> FD	<u>ES</u> FD	<u>ES</u> FD
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples	<u>Grab</u> Comp. # subsamples	<u>Grab</u> Comp. # subsamples
Sample Time	<u>1020</u>	<u>1021</u>	<u>1022</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		
	Entered Validated	Entered Validated	Entered Validated

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3022</u>	<u>1R8-3023</u>	<u>1R8-3024</u>
Location ID	<u>TEL 12110102 G-08 GC.7</u>	<u>TEL 12110102 G-08 GC.7</u>	<u>TEL 12110102 G-08 GC.7</u>
Sample Group	<u>G-08</u>	<u>G-08</u>	<u>G-08</u>
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>ES</u> FD _____	<u>ES</u> FD _____	<u>ES</u> FD _____
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1030</u>	<u>1031</u>	<u>1032</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		
Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

**LIBBY SISTER SITES FIELD SAMPLE DATA SHEET**  
**SOIL-LIKE MATERIALS**

Scenario No.: N/A Field Logbook No: 1 Page No:        Sampling Date: 10/15/02  
Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
Land Use: (circle) Residential School Commercial Mining Roadway Other Industrial  
Sampling Team: (circle) CDM PES Other:        Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3025</u>	<u>1R8-3026</u>	<u>1R8-3027</u>
Location ID	<u>TEL 121402 ET8617</u>	<u>TEL 121402 ET8617</u>	<u>TEL 121402 ET8617</u>
Sample Group			
Location Description (circle) <u>ES - Eldrie Salts</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples <u>      </u>	<u>Grab</u> Comp. # subsamples <u>      </u>	<u>Grab</u> Comp. # subsamples <u>      </u>
Sample Time	<u>1035</u>	<u>1036</u>	<u>1037</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>	<u>NPO</u>	<u>Trace of Product</u>
	Entered Validated	Entered Validated	Entered Validated

Sheet No.: S- SL-10

**LIBBY SISTER SITES FIELD SAMPLE DATA SHEET**  
**SOIL-LIKE MATERIALS**

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3028</u>	<u>IR8-3029</u>	<u>IR8-3030</u>
Location ID	<u>TEC 12nd St 17th G16</u>	<u>TEC 12nd St 17th G16</u>	<u>TEC 12nd St 17th G16</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1040</u>	<u>1041</u>	<u>1042</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		
Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other: \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3031</u>	<u>IR8-3032</u>	<u>IR8-3033</u>
Location ID	<u>121022-16 G15</u>	<u>121022-16 G15</u>	<u>121022-16 G15</u>
Sample Group			
Location Description (circle)	<u>ES - Eldred Substation</u> <u>PL - Parking Lot</u> Yard Soil Garden Soil Play Area Driveway Other: _____	Yard Soil Garden Soil Play Area Driveway Other: _____	Yard Soil Garden Soil Play Area Driveway Other: _____
C (circle)	FS FD _____	FS FD _____	FS FD _____
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other: _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other: _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other: _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1050</u>	<u>1051</u>	<u>1052</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>Product</u> →		<u>Abundant Product to 3.5' visual</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____



Sheet No.: S- SL-12

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3034</u>	<u>IR8-3035</u>	<u>IR8-3036</u>
Location ID	<u>TEC 1211402 G-15 G14</u>	<u>TEC 1211402 G-15 G14</u>	<u>TEC 1211402 G-15 G14</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	FS FD	FS FD	FS FD
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1100</u>	<u>1101</u>	<u>1102</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>	<u>NPO</u>	<u>Trace of Product Refused @ 2'</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3037</u>	<u>IR8-3038</u>	<u>IR8-3039</u>
Location ID	<u>14 G13</u>	<u>14 G13</u>	<u>14 G13</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	<u>FS</u> FD _____	<u>FS</u> FD _____	<u>FS</u> FD _____
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1110</u>	<u>1111</u>	<u>1112</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>	<u>Trace ?</u>	<u>NPO</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

Sheet No.: S- SL-14

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other Industrial  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3040</u>	<u>1R8-3041</u>	<u>1R8-3042</u>
Location ID	<u>TEC 211045 E-13 G12</u>	<u>TEC 211045 E-13 G12</u>	<u>TEC 211045 E-13 G12</u>
Sample Group			
Location Description (circle)	<u>ES - Electric Substation</u> <u>PL - Parking Lot</u> Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> FD	<u>FS</u> FD	<u>FS</u> FD
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples	<u>Grab</u> Comp. # subsamples	<u>Grab</u> Comp. # subsamples
Sample Time	<u>1120</u>	<u>1121</u>	<u>1122</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		
Entered	Validated	Entered	Validated

Sheet No.: S- SL-15

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other Industrial  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3043</u>	<u>IR8-3044</u>	<u>IR8-3045</u>
Location ID	<u>TEC slide 611</u>	<u>TEC slide 611</u>	<u>TEC slide 611</u>
Sample Group			
Location Description (circle) <u>ES - Eldrie Salts</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1125</u>	<u>1126</u>	<u>1127</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>	<u>Trace</u>	<u>Trace</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

Sheet No.: S- SL-16

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3046</u>	<u>IR8-3047</u>	<u>IR8-3048</u>
Location ID	<u>G 10</u>	<u>G10</u>	<u>G10</u>
Sample Group			
Location Description (circle)	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> FD _____	<u>FS</u> FD _____	<u>FS</u> FD _____
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1130</u>	<u>1131</u>	<u>1132</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3049</u>	<u>1R8-3050</u>	<u>1R8-3051</u>
Location ID	<u>G20</u>	<u>G20</u>	<u>G20</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	<u>ES</u> FD _____	<u>ES</u> FD _____	<u>ES</u> FD _____
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1140</u>	<u>1141</u>	<u>1147</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		<u>→</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

Sheet No.: S- SL-18

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No.: 1 Page No.: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3052</u>	<u>1R8-3053</u>	<u>1R8-3054</u>
Location ID	<u>G 21</u>	<u>G 21</u>	<u>G 21</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	FS <u>FD</u>	FS <u>FD</u>	FS <u>FD</u>
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1150</u>	<u>1151</u>	<u>1152</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NP 0</u>		<u>→</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

Sheet No.: S- SL-19

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3055</u>	<u>1R8-3056</u>	<u>1R8-3057</u>
Location ID	<u>G22</u>	<u>G22</u>	<u>G22</u>
Sample Group			
Location Description (circle) <u>ES - Eldrie Salton</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1155</u>	<u>1156</u>	<u>1157</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____



Sheet No.: S- SL-20

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3058</u>	<u>1R8-3059</u>	<u>1R8-3060</u>
Location ID	<u>G-23</u>	<u>G23</u>	<u>G23</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1200</u>	<u>1201</u>	<u>1202</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		<u>→</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other Industrial  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3061</u>	<u>1R8-3062</u>	<u>1R8-3063</u>
Location ID	<u>G24</u>	<u>G24</u>	<u>G24</u>
Sample Group			
Location Description (circle) <u>ES - Eldridge Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	<u>FS</u> FD _____	<u>FS</u> FD _____	<u>FS</u> FD _____
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other _____	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other _____	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1220</u>	<u>1221</u>	<u>1222</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

**LIBBY SISTER SITES FIELD SAMPLE DATA SHEET**  
**SOIL-LIKE MATERIALS**

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
Land Use: (circle) Residential School Commercial Mining Roadway Other Industrial  
Sampling Team: (circle) CDM PES Other: \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3064</u>	<u>IR8-3065</u>	<u>IR8-3066</u>
Location ID	<u>G25</u>	<u>G25</u>	<u>G25</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> FD	<u>FS</u> FD	<u>FS</u> FD
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples	<u>Grab</u> Comp. # subsamples	<u>Grab</u> Comp. # subsamples
Sample Time	<u>1240</u>	<u>1241</u>	<u>1242</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>Product Observed</u>	<u>Product Observed</u>	<u>Abundant Product @ 2'</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3067</u>	<u>1R8-3068</u>	<u>1R8-3069</u>
Location ID	<u>G26</u>	<u>G26</u>	<u>G26</u>
Sample Group			
Location Description (circle) <u>ES - Eldredge Station</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	FS FD	FS FD	FS FD
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1250</u>	<u>1251</u>	<u>1252</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u> →		
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

Sheet No.: S- 5L-24

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other: \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3070</u>	<u>1R8-3071</u>	<u>1R8-3072</u>
Location ID	<u>G27</u>	<u>G27</u>	<u>G27</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1255</u>	<u>1256</u>	<u>1257</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		<u>D</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3073</u>	<u>1R8-3074</u>	<u>1R8-3075</u>
Location ID	<u>G37</u>	<u>G37</u>	<u>G37</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	<u>ES</u> FD _____	<u>ES</u> FD _____	<u>ES</u> FD _____
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1420</u>	<u>1421</u>	<u>1422</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments			
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3076</u>	<u>1R8-3077</u>	<u>1R8-3078</u>
Location ID	<u>6-36</u>	<u>6-36</u>	<u>6-36</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	FS FD	FS FD	FS FD
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1440</u>	<u>1441</u>	<u>1442</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		
Entered	Validated	Entered	Validated

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3079</u>	<u>1R8-3080</u>	<u>1R8-3081</u>
Location ID	<u>G35</u>	<u>G35</u>	<u>G35</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other _____	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other _____	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1450</u>	<u>1451</u>	<u>1452</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>	<u>Product Visible</u> →	
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____



Sheet No.: S- SL-28

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3082</u>	<u>1R8-3083</u>	<u>1R8-3084</u>
Location ID	<u>G34</u>	<u>G34</u>	<u>G34</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1455</u>	<u>1456</u>	<u>1457</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location	<u>1455 PM</u>		
Field Comments	<u>N/A</u>		
Entered	Validated	Entered	Validated

Sheet No.: S- SL-29

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3085</u>	<u>1R8-3086</u>	<u>1R8-3087</u>
Location ID	<u>G-33</u>	<u>G-33</u>	<u>G-33</u>
Sample Group			
Location Description (circle) <u>ES - Eldred Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	FS FD	FS FD	FS FD
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1500</u>	<u>1501</u>	<u>1502</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>	<u>trace</u> →	
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

Sheet No.: S- SL-30

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other Industrial  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3088</u>	<u>1R8-3089</u>	<u>1R8-3089D</u>
Location ID	<u>G-32</u>	<u>G-32</u>	<u>G-32</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1510</u>	<u>1511</u>	<u>1512</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>	<u>trace</u> →	
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
Land Use: (circle) Residential School Commercial Mining Roadway Other: Industrial  
Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3091</u>	<u>1R8-3092</u>	<u>1R8-3093</u>
Location ID	<u>G-31</u>	<u>G-31</u>	<u>G-31</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
C (circle)	<u>FS</u> FD _____	<u>FS</u> FD _____	<u>FS</u> FD _____
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1520</u>	<u>1521</u>	<u>1522</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments	<u>NPO</u>		<u>7</u>
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

Sheet No.: S- SL-32

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp.)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3094</u>	<u>1R8-3095</u>	<u>1R8-3096</u>
Location ID	<u>G-30</u>	<u>G30</u>	<u>G30</u>
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>	<u>FS</u> <u>FD</u>
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other	<u>Mining Waste</u> <u>Subsurface Soil</u> Surface Soil Fill Other
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1530</u>	<u>1531</u>	<u>1532</u>
Top Depth (in.)	<u>0"</u>	<u>2"</u>	<u>6"</u>
Bottom Depth (in.)	<u>2"</u>	<u>6"</u>	<u>12"</u>
Map Location			
Field Comments			
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

**LIBBY SISTER SITES FIELD SAMPLE DATA SHEET**  
**SOIL-LIKE MATERIALS**

Scenario No.: N/A Field Logbook No: 1 Page No: \_\_\_\_\_ Sampling Date: 10/15/02  
 Address: 333 West 100 South Owner: Utah Power (Pacific Corp)  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Industrial)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>IR8-3100</u>	<u>IR8-3</u>	<u>IR8-3</u>
Location ID	<u>G04</u>		
Sample Group			
Location Description (circle) <u>ES - Electric Substation</u> <u>PL - Parking Lot</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>	Yard Soil Garden Soil Play Area Driveway <u>Other</u>
Category (circle)	<u>FS</u> FD _____	FS FD _____	FS FD _____
Matrix Type (circle)	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____	Mining Waste <u>Subsurface Soil</u> Surface Soil Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time			
Top Depth (in.)	<u>36"</u>	<u>2"</u>	<u>4"</u>
Bottom Depth (in.)	<u>48"</u>	<u>4"</u>	<u>12"</u>
Map Location			
Field Comments			
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

# LIBBY SISTER SITES FIELD SAMPLE DATA SHEET

## SOIL-LIKE MATERIALS

Scenario No.: \_\_\_\_\_ Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_ Sampling Date: 10/15  
 Address: 147 South 400 West Owner: Utah Power  
 Land Use: (circle) Residential School Commercial Mining Roadway Other (Utah Power)  
 Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: F. Morris

Data Item	Sample 1	Sample 2	Sample 3
Index ID	<u>1R8-3097</u>	<u>1R8-3098</u>	<u>1R8-3099</u>
Location ID			
Sample Group			
Location Description (circle)	Yard Soil Garden Soil Play Area Driveway Other <u>ES</u>	Yard Soil Garden Soil Play Area Driveway Other <u>ES</u>	Yard Soil Garden Soil Play Area Driveway Other <u>ES</u>
Category (circle)	<u>FS</u> FD _____	<u>FS</u> FD _____	<u>FS</u> FD _____
Matrix Type (circle)	<u>Mining Waste</u> <u>Subsurface Soil</u> <u>Surface Soil</u> Fill Other _____	<u>Mining Waste</u> <u>Subsurface Soil</u> <u>Surface Soil</u> Fill Other _____	<u>Mining Waste</u> <u>Subsurface Soil</u> <u>Surface Soil</u> Fill Other _____
Type (circle)	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____	<u>Grab</u> Comp. # subsamples _____
Sample Time	<u>1610</u>	<u>1615</u>	<u>1630</u>
Top Depth (in.)	<u>0" → 9" dug</u>	<u>0"</u>	<u>0"</u>
Bottom Depth (in.)	<u>2"</u>	<u>2"</u>	<u>2"</u>
Map Location	<u>lower level</u>		<u>100 S.</u>
Field Comments			
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

Sheet No: D-\_\_\_\_\_

# **SALT LAKE CITY, UTAH SITE INVESTIGATION FIELD SAMPLE DATA SHEET FOR PERSONAL AIR**

Employee Name: Frank MorrisSSN: XXX-XX-5789Task Performed: Soil sample collectionSite Visit Date: 10/15/02Sampling Team: PETRAK

Data Item	Cassette 1 <small>10/15/02</small>	Cassette 2	Cassette 3	Cassette 4
Field ID Number	LSS-457-UTSL -PA-5789			
Index ID	2K8-2011			
Category (circle)	FS Rep _____ Blank	FS Rep _____ Blank	FS Rep _____ Blank	FS Rep _____ Blank
Matrix Type	Indoor <u>Outdoor</u>	Indoor Outdoor	Indoor Outdoor	Indoor Outdoor
Location Description	SHOULDER			
Flow Meter Type	ROTAMETER			
Flow Meter ID No.	HF-1			
Pump ID Number	512001			
Start Date	10/15/02			
Start Time	0912 / 1419			
Start Flow (L/min)	1.88 / 1.88			
Stop Date	10/15/02			
Stop Time	1306 / 1635			
Stop Flow (L/min)	1.88 / 1.88			
Pump fault?	(No) Yes	No Yes	No Yes	No Yes
MET Station onsite?	(No) Yes	No Yes	No Yes	No Yes
Field Comments	ave = 1.88 Lpm x 234 min = 440 L  ave = 1.88 Lpm x 136 min = 256 L  = 696 L			

= 696 L



# SALT LAKE CITY, UTAH INVESTIGATION FIELD SAMPLE DATA SHEET FOR AIR

Address or Location ID: 147 S 400 W

GPS (if no address available): Northing \_\_\_\_\_ Easting \_\_\_\_\_

Owner: UTAH LIGHT & POWERLand Use Category: Residential School Commercial Mining Other (\_\_\_\_\_)Site Visit Date: 10/15/02Sampling Team: PETRAK

Data Item	Cassette 1	Cassette 2	Cassette 3	Cassette 4
Field ID Number	<del>LSS-1011-UTSL</del> -AA-GP1			
Index ID	218-2012			
Category (circle)	<u>FS</u> Rep _____ Blank	FS Rep _____ Blank	FS Rep _____ Blank	FS Rep _____ Blank
Matrix Type	Indoor <u>Outdoor</u>	Indoor Outdoor	Indoor Outdoor	Indoor Outdoor
Location Description	GEOPROBE TOOL SHELF			
Flow Meter Type	POTAMETER			
Flow Meter ID No.	HF-1			
Pump ID Number	508843			
Start-Date	10/15/02			
Start-Time	0910			
Start-Flow (L/min)	1.88			
Stop-Date	10/15/02			
Stop-Time	1616			
Stop-Flow (L/min)	1.88			
Pump fault?	<u>(No)</u> Yes No Yes	No Yes	No Yes	No Yes
MET Station onsite?	<u>(No)</u> Yes No Yes	No Yes	No Yes	No Yes
Field Comments	Flow = 1.88 Lpm 2426 min = 801 L			

# SALT LAKE CITY, UTAH INVESTIGATION FIELD SAMPLE DATA SHEET FOR AIR

Address or Location ID: 733 W 800 S

GPS (if no address available): Northing \_\_\_\_\_ Easting \_\_\_\_\_

Owner: Scott SimonLand Use Category: Residential School Commercial Mining Other (\_\_\_\_\_)Site Visit Date: 10/15/02Sampling Team: PETRAK

Data Item	<sup>10/15/02</sup> Cassette 1	Cassette 2	Cassette 3	Cassette 4
Field ID Number	<del>LS-UTSL-BK-5003</del> <del>LS-UTSL-5003</del>	LS-UTSL-BK1	LS-UTSL-BK2	
Index ID	228-2014	228-2016	228-2017	
Category (circle)	<u>FS</u> Rep _____ Blank	FS Rep _____ <u>Blank</u>	FS Rep _____ <u>Blank</u>	FS Rep _____ Blank
Matrix Type	Indoor <u>Outdoor</u>	Indoor <u>Outdoor</u>	Indoor <u>Outdoor</u>	Indoor Outdoor
Location Description	GRAVEL LOT WEST END	BLANK	BLANK	
Flow Meter Type	ROTAMETER			
Flow Meter ID No.	HF-1			
Pump ID Number	0891			
Start-Date	10/15/02			
Start-Time	1428			
Start-Flow (L/min)	<sup>10/15/02</sup> 10.04			
Stop-Date	10/16/02			
Stop-Time	1658			
Stop-Flow (L/min)	10.04			
Pump fault?	<u>No</u> Yes No Yes No Yes No Yes	No Yes No Yes No Yes No Yes	No Yes No Yes No Yes No Yes	No Yes No Yes No Yes No Yes
MET Station onsite?	<u>No</u> Yes No Yes No Yes No Yes	No Yes No Yes No Yes No Yes	No Yes No Yes No Yes No Yes	No Yes No Yes No Yes No Yes
Field Comments	Flow = 10.04 Lpm x 150 min = 1506 L	↓	↓	

M. Petrak  
10/15/02

# SALT LAKE CITY, UTAH INVESTIGATION FIELD SAMPLE DATA SHEET FOR AIR

Address or Location ID: 147 S 400 W

GPS (if no address available): Northing \_\_\_\_\_ Easting \_\_\_\_\_

Owner: UTAH POWER & LIGHTLand Use Category: Residential School Commercial Mining Other (\_\_\_\_\_)Site Visit Date: 10/16/02Sampling Team: PETRAK

Data Item	Cassette 1	Cassette 2	Cassette 3	Cassette 4
Field ID Number	LSS-UTSL-AA-5005	LSS-UTSL-AA-5006	LSS-UTSL-AA-5007	LSS-UTSL-AA-5008
Index ID	2128-2018	2128-2019	2128-2020	2128-2021
Category (circle)	<u>FS</u> Rep _____ Blank	<u>FS</u> Rep _____ Blank	<u>FS</u> Rep _____ Blank	<u>FS</u> Rep _____ Blank
Matrix Type	Indoor <u>Outdoor</u>	Indoor <u>Outdoor</u>	Indoor <u>Outdoor</u>	Indoor <u>Outdoor</u>
Location Description	SE corner	SW corner, on low concrete wall	NE NW corner, near bank	NE corner
Flow Meter Type	ROTAMETER	ROTAMETER	ROTAMETER	ROTAMETER
Flow Meter ID No.	HF-1	HF-1	HF-1	HF-1
Pump ID Number	1007	6891	6899	0387
Start-Date	10/16/02	10/16/02	10/16/02	10/16/02
Start-Time	0805	0810	0814	0817
Start-Flow (L/min)	10.04	10.04	10.04	10.04
Stop-Date	10/16/02	10/16/02	10/16/02	10/16/02
Stop-Time	1219	1220	1222	1226
Stop-Flow (L/min)	9.78	10.04	10.04	9.78
Pump fault?	<u>No</u> Yes	<u>No</u> Yes	<u>No</u> Yes	<u>No</u> Yes
MET Station onsite?	<u>No</u> Yes	<u>No</u> Yes	<u>No</u> Yes	<u>No</u> Yes
Field Comments	ave = 9.91 Lpm x 254 min = 2517 L	ave = 10.04 Lpm x 250 min = 2510 L	ave = 10.04 Lpm x 248 min = 2490 L	ave = 9.91 Lpm x 249 min = 2468 L

**Attachment 3**

**Site Photographs**

# Color Photo(s)

The following pages  
contain color that does  
not appear in the  
scanned images.

To view the actual images, please  
contact the Superfund Records  
Center at (303) 312-6473.

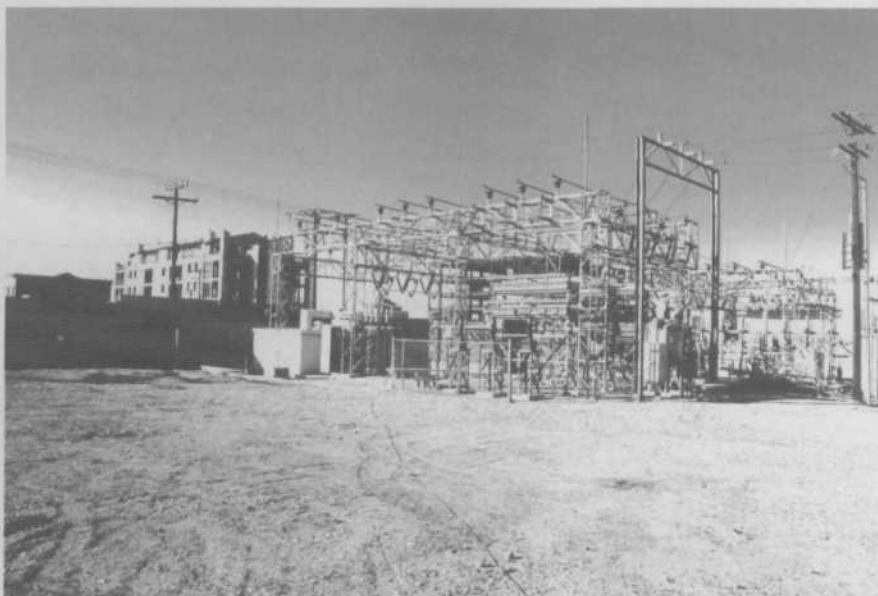


Photo 1. Southwest view of PacifiCorp electrical substation from air monitoring station S008.



Photo 2. Photograph facing west showing the location of former railroad spur that supplied vermiculite to former processing plant.



Photo 3. Photograph facing south showing site of former processing plant (SLC2).



Photo 4. Photograph facing northeast showing air monitoring station S008 in foreground. Note Delta Center in background.



Photo 5. Photograph facing south showing remaining foundation from former processing plant. Note air monitoring station S006 on wall.



Photo 6. Close up view of surface material (visible vermiculite mixed with gravel) at SLC2.





Photo 7. Photograph facing southwest showing air monitoring station S006. Note vermiculite product in foreground.

**Attachment 4**  
**Analytical Data**

Table 1. All sample analytical data

Index ID	Grid Location	Depth (inches)	X Coordinate	Y Coordinate	PLM Result (% LA)	Visible Vermiculite
1R8-3001	G00	0-2	1529160.55024000000	7448075.41724000000	< 1	No
1R8-3002	G00	2-6	1529160.55024000000	7448075.41724000000	ND	Trace
1R8-3003	G00	6-12	1529160.55024000000	7448075.41724000000	2	Trace
1R8-3004	G01	0-2	1529157.68500000000	7448101.66152000000	< 1	Trace
1R8-3005	G01	2-6	1529157.68500000000	7448101.66152000000	< 1	Trace
1R8-3006	G01	6-12	1529157.68500000000	7448101.66152000000	< 1	Trace
1R8-3007	G02	0-2	1529162.61407000000	7448129.49092000000	< 1	Trace
1R8-3008	G02	2-6	1529162.61407000000	7448129.49092000000	< 1	Yes
1R8-3009	G02	6-12	1529162.61407000000	7448129.49092000000	< 1	Yes
1R8-3010	G03	0-2	1529160.74697000000	7448152.29260000000	< 1	No
1R8-3011	G03	2-6	1529160.74697000000	7448152.29260000000	< 1	No
1R8-3012	G03	6-12	1529160.74697000000	7448152.29260000000	3	Abundant to 4 ft
1R8-3013	G04	0-2	1529159.13335000000	7448174.41441000000	3	No
1R8-3014	G04	2-6	1529159.13335000000	7448174.41441000000	12	Abundant to 4 ft
1R8-3015	G04	6-12	1529159.13335000000	7448174.41441000000	15	Abundant to 4 ft
1R8-3100	G04	36-42	1529159.13335000000	7448174.41441000000	18	Visible to 9.5 ft
1R8-3016	G05	0-2	1529159.08742000000	7448199.97886000000	< 1	No
1R8-3017	G05	2-6	1529159.08742000000	7448199.97886000000	4	Abundant
1R8-3018	G05	6-12	1529159.08742000000	7448199.97886000000	2	No
1R8-3019	G06	0-2	1529158.09646000000	7448227.69226000000	< 1	No
1R8-3020	G06	2-6	1529158.09646000000	7448227.69226000000	2	No
1R8-3021	G06	6-12	1529158.09646000000	7448227.69226000000	< 1	No
1R8-3022	G07	0-2	1529157.81215000000	7448253.20613000000	< 1	No
1R8-3023	G07	2-6	1529157.81215000000	7448253.20613000000	< 1	No
1R8-3024	G07	6-12	1529157.81215000000	7448253.20613000000	< 1	No
1R8-3025	G17	0-2	1529183.18065000000	7448249.28638000000	< 1	No
1R8-3026	G17	2-6	1529183.18065000000	7448249.28638000000	< 1	No
1R8-3027	G17	6-12	1529183.18065000000	7448249.28638000000	< 1	Trace
1R8-3028	G16	0-2	1529184.20313000000	7448226.34281000000	< 1	No
1R8-3029	G16	2-6	1529184.20313000000	7448226.34281000000	< 1	No
1R8-3030	G16	6-12	1529184.20313000000	7448226.34281000000	< 1	No
1R8-3031	G15	0-2	1529183.19428000000	7448198.20745000000	< 1	Yes
1R8-3032	G15	2-6	1529183.19428000000	7448198.20745000000	< 1	Yes
1R8-3033	G15	6-12	1529183.19428000000	7448198.20745000000	12	Abundant to 3.5 ft
1R8-3034	G14	0-2	1529184.39506000000	7448179.01794000000	< 1	No
1R8-3035	G14	2-6	1529184.39506000000	7448179.01794000000	< 1	No
1R8-3036	G14	6-12	1529184.39506000000	7448179.01794000000	< 1	Trace
1R8-3037	G13	0-2	1529183.81120000000	7448152.02845000000	< 1	No
1R8-3038	G13	2-6	1529183.81120000000	7448152.02845000000	< 1	Trace
1R8-3039	G13	6-12	1529183.81120000000	7448152.02845000000	< 1	No

Table 1. All sample analytical data

Index ID	Grid Location	Depth (Inches)	X Coordinate	Y Coordinate	PLM Result (% LA)	Visible Vermiculite
1R8-3040	G12	0-2	1529183.76803000000	7448127.74895000000	< 1	No
1R8-3041	G12	2-6	1529183.76803000000	7448127.74895000000	< 1	No
1R8-3042	G12	6-12	1529183.76803000000	7448127.74895000000	ND	No
1R8-3043	G11	0-2	1529184.10749000000	7448104.99355000000	< 1	No
1R8-3044	G11	2-6	1529184.10749000000	7448104.99355000000	< 1	Trace
1R8-3045	G11	6-12	1529184.10749000000	7448104.99355000000	< 1	No
1R8-3046	G10	0-2	1529182.31359000000	7448075.84866000000	< 1	No
1R8-3047	G10	2-6	1529182.31359000000	7448075.84866000000	< 1	No
1R8-3048	G10	6-12	1529182.31359000000	7448075.84866000000	< 1	No
1R8-3049	G20	0-2	1529207.87188000000	7448082.59448000000	< 1	No
1R8-3050	G20	2-6	1529207.87188000000	7448082.59448000000	< 1	No
1R8-3051	G20	6-12	1529207.87188000000	7448082.59448000000	< 1	No
1R8-3052	G21	0-2	1529204.60104000000	7448094.54059000000	< 1	No
1R8-3053	G21	2-6	1529204.60104000000	7448094.54059000000	< 1	No
1R8-3054	G21	6-12	1529204.60104000000	7448094.54059000000	< 1	No
1R8-3055	G22	0-2	1529208.30691000000	7448126.47141000000	< 1	No
1R8-3056	G22	2-6	1529208.30691000000	7448126.47141000000	< 1	No
1R8-3057	G22	6-12	1529208.30691000000	7448126.47141000000	< 1	No
1R8-3058	G23	0-2	1529208.49647000000	7448153.17253000000	< 1	No
1R8-3059	G23	2-6	1529208.49647000000	7448153.17253000000	< 1	No
1R8-3060	G23	6-12	1529208.49647000000	7448153.17253000000	< 1	No
1R8-3061	G24	0-2	1529208.99842000000	7448178.02983000000	< 1	No
1R8-3062	G24	2-6	1529208.99842000000	7448178.02983000000	< 1	No
1R8-3063	G24	6-12	1529208.99842000000	7448178.02983000000	< 1	No
1R8-3064	G25	0-2	1529208.21533000000	7448201.98425000000	< 1	Yes
1R8-3065	G25	2-6	1529208.21533000000	7448201.98425000000	< 1	Yes
1R8-3066	G25	6-12	1529208.21533000000	7448201.98425000000	1	Abundant
1R8-3067	G26	0-2	1529209.95078000000	7448228.71332000000	2	No
1R8-3068	G26	2-6	1529209.95078000000	7448228.71332000000	1	No
1R8-3069	G26	6-12	1529209.95078000000	7448228.71332000000	< 1	No

Table 1. All sample analytical data

Index ID	Grid Location	Depth (inches)	X Coordinate	Y Coordinate	PLM Result (% LA)	Visible Vermiculite
1R8-3070	G27	0-2	1529208.80332000000	7448251.13666000000	< 1	No
1R8-3071	G27	2-6	1529208.80332000000	7448251.13666000000	< 1	No
1R8-3072	G27	6-12	1529208.80332000000	7448251.13666000000	ND	No
1R8-3073	G37	0-2	1529233.07319000000	7448246.36924000000	< 1	No
1R8-3074	G37	2-6	1529233.07319000000	7448246.36924000000	< 1	No
1R8-3075	G37	6-12	1529233.07319000000	7448246.36924000000	ND	No
1R8-3076	G36	0-2	1529232.75949000000	7448226.50694000000	1	No
1R8-3077	G36	2-6	1529232.75949000000	7448226.50694000000	< 1	No
1R8-3078	G36	6-12	1529232.75949000000	7448226.50694000000	ND	No
1R8-3079	G35	0-2	1529232.53222000000	7448202.88689000000	< 1	No
1R8-3080	G35	2-6	1529232.53222000000	7448202.88689000000	< 1	Yes
1R8-3081	G35	6-12	1529232.53222000000	7448202.88689000000	< 1	Yes
1R8-3082	G34	0-2	1529232.66666000000	7448174.54218000000	7	No
1R8-3083	G34	2-6	1529232.66666000000	7448174.54218000000	ND	No
1R8-3084	G34	6-12	1529232.66666000000	7448174.54218000000	ND	No
1R8-3085	G33	0-2	1529232.09049000000	7448151.65707000000	< 1	No
1R8-3086	G33	2-6	1529232.09049000000	7448151.65707000000	< 1	Trace
1R8-3087	G33	6-12	1529232.09049000000	7448151.65707000000	< 1	Trace
1R8-3088	G32	0-2	1529231.37015000000	7448127.35525000000	ND	No
1R8-3089	G32	2-6	1529231.37015000000	7448127.35525000000	< 1	Trace
1R8-3090	G32	6-12	1529231.37015000000	7448127.35525000000	< 1	Trace
1R8-3091	G31	0-2	1529232.09060000000	7448102.05478000000	< 1	No
1R8-3092	G31	2-6	1529232.09060000000	7448102.05478000000	< 1	No
1R8-3093	G31	6-12	1529232.09060000000	7448102.05478000000	< 1	No
1R8-3094	G30	0-2	1529230.96560000000	7448071.52445000000	< 1	No
1R8-3095	G30	2-6	1529230.96560000000	7448071.52445000000	< 1	No
1R8-3096	G30	6-12	1529230.96560000000	7448071.52445000000	< 1	No
1R8-3097	Lower Level	0-2	1529147.24960000000	7448109.36813000000	< 1	No
1R8-3098	Upper Level	0-2	1529147.37447000000	7448118.23289000000	3	No
1R8-3099	Fmr RR Spur	0-2	1529159.42129000000	7448212.78079000000	< 1	No

All samples analyzed by PLM (NIOSH 9002) except for 1R8-3101 and 1R8-3102

PLM polarized light microscopy

% percent

LA Libby Amphibole

< less than

ND nondetect

Table 1 Air sample analytical data

Index ID	Air Sample Type Description	LA Fiber Count	Volume (L)	LA Concentration (S/cc)	X Coordinate	Y Coordinate
2R8-2011	Personal - Sampler	4	696	1.10E-02	NA	NA
2R8-2012	Stationary - tractor tool shelf	1	801	2.30E-03	NA	NA
2R8-2016	Blank	0	0	ND	NA	NA
2R8-2017	Blank	Archived	0	Archived	NA	NA
2R8-2018	Clearance - S005	0	2517	ND	1529207.400650000000	7448100.366560000000
2R8-2019	Clearance - S006	0	2510	ND	1529151.288220000000	7448108.366130000000
2R8-2020	Clearance - S007	0	2490	ND	1529101.328490000000	7448190.518820000000
2R8-2021	Clearance - S008	0	2468	ND	1529217.944360000000	7448230.559860000000

All samples analyzed by TEM/AHERA (NIOSH 7402)

LA Libby Amphibole  
L liters  
S/cc structures per cubic centimeter  
AHERA Asbestos Hazard Emergency Response Act  
TEM transmission electron microscopy  
NA not available  
ND nondetect

# Color Map(s)

The following pages  
contain color that does  
not appear in the  
scanned images.

To view the actual images, please  
contact the Superfund Records  
Center at (303) 312-6473.

Figure 2

Site Detail Map  
Libby Asbestos Project  
Sisters of Libby  
Salt Lake City, UT-SLC2

Legend

- ▲ Air Monitoring Station
- Approximate Boundary of  
Former Processing Building



CDM





Figure 3

Surface Soil Sample Results  
(0 to 2 Inches)

Libby Asbestos Project  
Sisters of Libby  
Salt Lake City, UT-SLC2

Legend  
PLM Analysis  
Tremolite-Actinolite %

- 0
- <=1
- 2 - 3
- 4
- >5



CDM

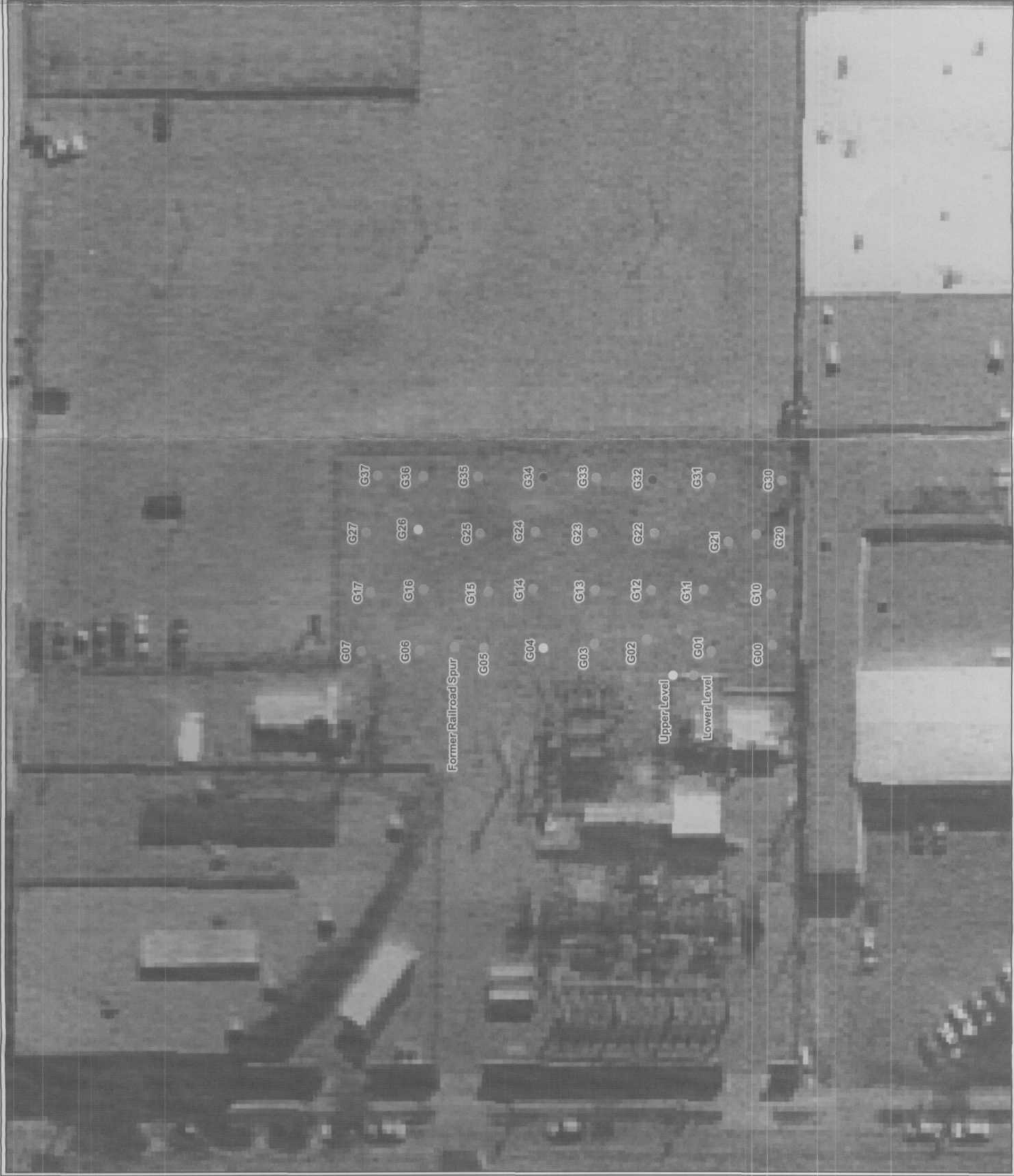


Figure 4

Subsurface Soil Sample Results  
(2 to 6 Inches)

Libby Asbestos Project  
Sisters of Libby  
Salt Lake City, UT-SLC2

Legend  
PLM Analysis  
Tremolite-Actinolite %

- 0
- <=1
- 2 - 3
- 4
- >5



CDM

Former Railroad Spur

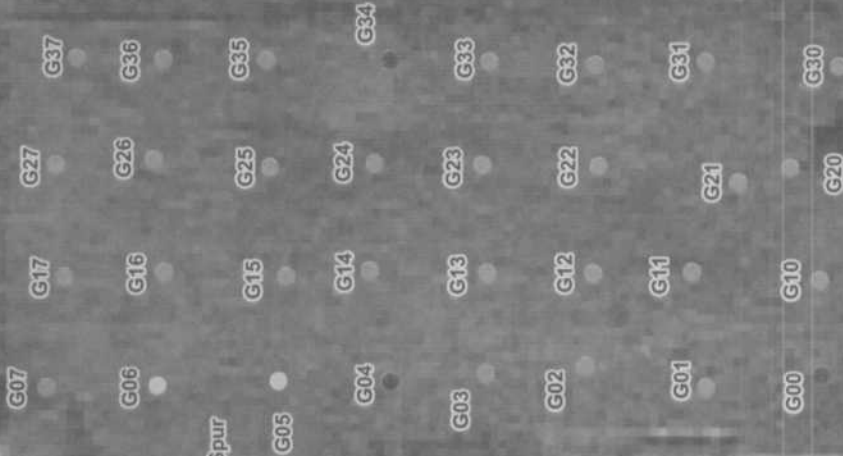


Figure 5

Subsurface Soil Sample Results  
(6 to 12 Inches)  
Libby Asbestos Project  
Sisters of Libby  
Salt Lake City, UT-SLC2

Legend  
PLM Analysis  
Tremolite-Actinolite %

- 0
- ≤1
- 2 - 3
- 4
- >5

Subsurface Soil (36 - 42 Inch Depth)

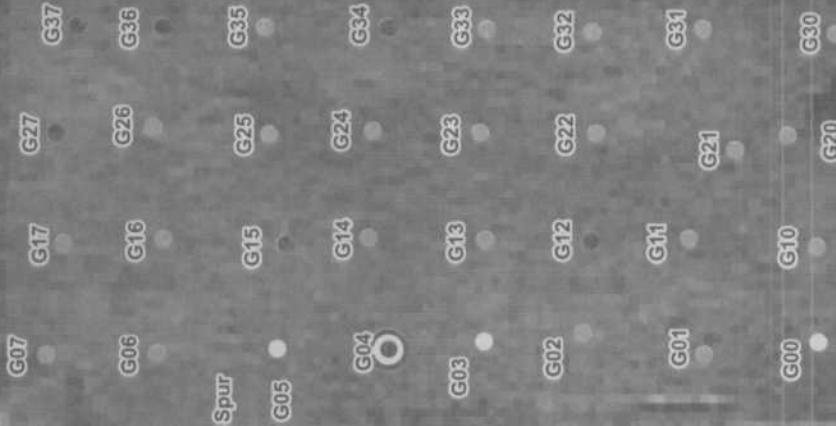
○ >5

Refusal at 9.5 feet, product full length  
(1 foot offset south of G04)



CDM

Former Railroad Spur





# Sampling and Analysis Plan

## Revision 1

**Former Vermiculite Intermountain Facility-SLC2**

**100 South 333 West**

**Salt Lake City, Utah**

*Libby Sister Sites (Asbestos Project)*

**July 2003**



*Prepared for:*



**U.S. EPA Region 8**  
999 18<sup>th</sup> Street  
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Denver, Colorado 80202-2466

*Prepared by:*



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Environmental Engineering Division, DTS-33  
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**CDM Federal Programs Corporation**  
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Cambridge, Massachusetts 02319

**Revision 1  
Sampling and Analysis Plan  
for  
Libby Sister Sites (Asbestos Project)  
Former Vermiculite Intermountain Facility-SLC2  
Salt Lake City, Utah**

**EPA Region VIII**

**July 2003**

**Contract No. DTRS57-99-D-00017  
Task Order No. C0023**

**Prepared for:**

**U.S. Environmental Protection Agency - Region VIII  
Emergency Response Office  
Denver, Colorado**

**Prepared by:**

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**and**

**CDM Federal Programs Corporation  
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50 Hampshire Street  
Cambridge, Massachusetts 02139**

Revision 1  
Sampling and Analysis Plan  
for  
Libby Sister Sites  
Former Vermiculite Intermountain Facility-SLC2  
Salt Lake City, Utah

EPA Region VIII

July 2003

Contract No. DTRS57-99-D-00017  
Task Order No. C0023

Prepared By: \_\_\_\_\_ Date: \_\_\_\_\_

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Approved By: \_\_\_\_\_ Date: \_\_\_\_\_

Floyd Nichols  
EPA On-Scene Coordinator

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# Acronyms

AHERA	Asbestos Hazard Emergency Response Act
ACM	asbestos-containing material
AIHA	American Industrial Hygiene Association
ASTM	American Society for Testing and Materials
CDM	CDM Federal Programs Corporation
CDM Inc.	CDM Incorporated
cm <sup>2</sup>	square centimeter
COC	chain-of-custody
DI	deionized
DPT	direct-push technology
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
GPS	global positioning system
IAG	interagency agreement
LA	Libby asbestos
L/min	liters per minute
µm	micron
MACTEC	MACTEC Companies
MCE	mixed cellulose ester
NESHAP	National Emission Standards for Hazardous Air Pollutants
NIOSH	National Institute of Occupational Safety and Health
NIST	National Institute of Standards and Technology
NPL	National Priorities List
OSC	on-site coordinator
OSHA	Occupational Safety and Health Administration
PCBs	polychlorinated biphenyls
PCM	phase contrast microscopy
MACTEC	MACTEC Companies
PLM	polarized light microscopy
RA	removal assessment
SAP	sampling and analysis plan
SDG	sample delivery group
SHSP	site health and safety plan
SLC2	Salt Lake City #2 (original site)
SOP	standard operating procedure
TEM	transmission electron microscopy
USGS	U.S. Geological Survey
Volpe Center	John A. Volpe National Transportation Systems Center
°F	degrees Fahrenheit

# Section 1

## Introduction

The U.S. Department of Transportation's John A. Volpe National Transportation Systems Center (Volpe Center) has an Interagency Agreement (IAG) with the U.S. Environmental Protection Agency (EPA) Region VIII for environmental engineering and related support.

Since November 1999, the Environmental Engineering Division (DTS-33) of the Volpe Center has been providing EPA Region VIII with immediate environmental engineering and site assessment support at Libby, MT. The Volpe Center, its contractor CDM Federal Programs Corporation (CDM), and CDM's subcontractor MACTEC Companies (MACTEC), have been requested by EPA Region VIII to conduct walk-through site visits and limited sampling activities to support removal assessment (RA) reporting at two locations within Salt Lake City. These locations were identified, via the U.S. Geological Survey (USGS) or Bureau of Mines publications, to have received asbestos ore or vermiculite from Libby, Montana. Each of the sites has performed either small batch exfoliation, used vermiculite as part of a manufacturing process, or sold vermiculite.

This sampling and analysis plan (SAP) defines sampling and analytical procedures that will be used for conducting expanded media sampling at the former Vermiculite Intermountain facility SLC2 in the vicinity of 333 West 100 South, Salt Lake City, Utah.

### 1.1 Project Objectives

The objectives of the Libby Sister Sites (Region VIII) project are to:

- 1) Determine if any potential sources of Libby Amphibole (LA) (tremolite/actinolite series) asbestos contamination are present at the site related to the processing of vermiculite ore. Potential sources to be investigated include soil, waste/product, dust inside buildings, and ambient air.
- 2) Document any observed evidence of vermiculite product or other related waste with detailed notes and digital photographs.

Determine the vertical and horizontal extent of asbestos contamination.

## 1.2 Project Organization and Responsibilities

Organization and responsibilities specific to this field investigation are listed in this section. For this data collection effort, key management personnel are as follows:

<u>Individual</u>	<u>Role</u>
Floyd Nichols	EPA On-Scene Coordinator (OSC)
John McGuiggin	Volpe Center Project Manager
Paul Kudarauskas	Volpe Center Field Team Member
Tim Wall	CDM Task Order Manager
Frank Morris	CDM Task Leader
Tommy Cook	CDM Field Team Member (soils/waste/product)
Jennifer Oxford	CDM Quality Assurance Coordinator
Brian Stewart	MACTEC Task Leader
Melissa Petrak	MACTEC Field Team Member (dust/air)

The entire field and data gathering effort will be conducted by a team consisting of one CDM member who will be responsible for the soil and waste/product sampling and one MACTEC member in charge of the dust sample collection and ambient/personal air sampling. The team will have a designated Volpe Center team leader. The contractor team lead will have prior experience with performing similar activities under the EPA Region VIII Libby Asbestos Project. A Volpe Center and/or EPA representative will accompany the field team and will work with contractor personnel to determine the site-specific sampling requirements for each site.

## Section 2

# Project Background

### 2.1 Source of the Vermiculite

The Town of Libby is located in the extreme northwest corner of Montana. According to historical mining records, up to 80 percent of the world's vermiculite has come from the W.R. Grace Vermiculite Mine located on Zonolite Mountain approximately seven miles northeast of Libby. Vermiculite is a mineral that is used in various building materials and textiles. Disseminated within the enormous deposit of vermiculite on Zonolite Mountain is the mineral tremolite, a rare and exceedingly toxic form of asbestos. Over the approximately 60-year life of the mine, tremolite asbestos was released into the environment as a by-product of the mining and ore-processing activities.

The Zonolite Mine began operation in 1924 by owner Edward Alley. In 1925, Great Northern Railroad shipped the first boxcar of "zonolite" from Libby to an Ohio company that used it to insulate bank vaults, office safes, and filing cabinets. Other firms used the material to make building boards and roofing materials. Processing the material was straightforward. The vermiculite ore was stripped from the mine and hauled in trucks to a mill, where it was separated into various commercial sizes through a screening system. Some of the ore was shipped untouched. Other material was sent to an expansion plant where it was run through ovens at about 2,000 degrees Fahrenheit (°F), causing the material to expand to 15 times its original size. In 1939, Alley's Zonolite Mine merged with another mining company that eventually became known as the Zonolite Co.

In 1963, the company was sold to W.R. Grace and Co. who expanded the operation and increased production. Through the '60s, '70s, and '80s, millions of tons of Libby vermiculite ore were shipped by rail to numerous processing plants in 30 states and six foreign countries.

### 2.2 Environmental Setting

EPA has determined that the vermiculite ore mined from the mountains surrounding Libby, MT is contaminated with LA asbestos. The ore was shipped throughout the United States both as a processed and unprocessed material. The EPA has been conducting various investigations to determine other potentially contaminated properties (outside of Libby), which may have been impacted by the Libby mining operations. In support of these investigations, the Volpe Center has been requested by EPA Region VIII to conduct an assessment at the former Vermiculite Intermountain facility in Salt Lake City, CO. This location was identified by USGS and Bureau of Mines publications as a site that received ore or vermiculite from Libby, MT. Each of the sites requiring further investigation either performed small batch

exfoliation, used vermiculite as part of a manufacturing process, or sold vermiculite directly.

This SAP defines sampling and analytical procedures that will be used for conducting additional media sampling at the former Vermiculite Intermountain facility in Salt Lake City, Utah. The site where the former plant was located is now owned by Utah Power (Pacific Corporation). The footprint of the now demolished popping plant and former railroad spur are adjacent to and overlap onto an electrical substation.

## 2.3 Previous Investigations

### 2.3.1 Site Description/Known History

SLC2 is located at 100 South 330 West just south of the Delta Center in downtown Salt Lake City. The site is situated between a power transfer station and an asphalt parking lot. Site detail of SLC2 is illustrated on Figure 2-1. The aerial photograph shown in the figure was taken in 2000 from digital imagery obtained from Olympus Aerial Surveys, Inc. According to historical records, SLC2 was the original location for the Intermountain Insulation Company (formerly Vermiculite Intermountain) processing facility. The site (area shown in red) is bordered on the north by 100 South Street and Artistic Printing Company, on the west by 400 West Street, on the south by Utah Paperbox Company, and on the east by an asphalt parking lot leased by AMPCO. The former processing facility is now demolished and the site is currently owned in part by Pacific Corporation, a parent company of Utah Power and Light. The original plant boundaries are shown in green.

Historical research conducted by the EPA On-Scene Coordinator prior to any sampling activities indicated that Intermountain Insulation had operated at this site from about 1940 to 1984 before relocating their operations to another site at 733 West 800 South (SLC1). Intermountain Insulation, under license to W.R. Grace Construction Products Division, manufactured and distributed insulation, fireproofing, vermiculite soil conditioner, masonry fill and concrete plaster aggregate until the company went bankrupt in 1987.

The exfoliation facility was formerly known as Vermiculite Intermountain. The company later changed its name to Intermountain Insulation (date unknown). Vermiculite-containing material was shipped to SLC2 via railcars. According to interviews with a previous employee, the material was scattered about the property due to leakage from standing rail cars and from the actual transfer of the material from the railcars to the processing plant.

The original site work, which involved surface and subsurface soil sampling and baseline ambient air sampling, revealed up to 3% LA on the ground surface and up to 18% LA in the subsurface. The investigation was conducted by Ms. Joyce Ackerman (EPA), Mr. Paul Kudarauskas (Volpe Center), Mr. Frank Morris (CDM), and Melissa Petrak (MACTEC) on October 14 through October 16, 2002. The findings are reported in a summary report (CDM 2003a), which will be incorporated into the subsequent focused RA Report. A proactive surface cleanup was reported by the property owner

focused RA Report. A proactive surface cleanup was reported by the property owner (Pacific Corporation) based on results of this initial investigation during the fall of 2002. Written documentation of this effort is currently unavailable. However, the site visit on May 29, 2003 revealed visible contamination still present in the reportedly affected areas.

## 2.4 Contaminant of Concern

The only potential contaminant of concern being investigated at this site is asbestos, specifically the amphibole minerals from the Libby, MT mine. Asbestos fibers are odorless and tasteless and vary in length, structure, and chemical composition. Fibers are microscopic and environmentally persistent. They do not evaporate, burn or dry out from heat, or erode in water. Toxicity of different type fibers varies, but exposure to any one of them can be fatal. Libby amphiboles (LA), especially tremolite and actinolite, are considered by many to be the most toxic.

The human health risks associated with asbestos fibers released in the environment include:

- Malignant mesothelioma, a cancer of the pleural or peritoneal cavity. In early stages of the disease, cancer is found in the lining of the chest cavity near the lung and heart or in the diaphragm. Mesothelioma may spread to tissue surrounding the lungs or other organs. Virtually all mesothelioma cases are attributable to asbestos exposure.
- Asbestosis, the scarring of the tissue of the lung itself from inhalation of fibers. It ranges in severity from mild impairment to disabling and eventually fatal.

Asbestos and smoking both cause lung cancer, but a population with a history of smoking combined with exposure to asbestos creates a much higher risk of developing asbestos-related diseases.

PCB levels from biased soil samples are also being considered because the site is associated with an electrical substation. However, results will only be evaluated at this time to see if PCBs are present and if they would be a potential problem for removal and/or land disposal of soils.



## Section 3

# Data Quality Objectives

To ensure that data of sufficient quality and quantity are collected to meet project objectives, the data quality objective (DQO) process (EPA 2000) was utilized to develop DQOs for the soil, waste/product, dust, and air sampling tasks. The DQO process is a series of steps based on the scientific method that are designed to ensure that the type, quantity, and quality of environmental data used in decision making are appropriate for the intended purpose. The DQO process consists of the following seven steps:

- |         |   |
|---------|---|
| Step 1: | State the Problem;                      |
| Step 2: | Identify the Decision;                  |
| Step 3: | Identify Inputs to the Decision;        |
| Step 4: | Define the Study Boundaries;            |
| Step 5: | Develop a Decision Rule;                |
| Step 6: | Specify Limits on Decision Errors; and  |
| Step 7: | Optimize the Design for Obtaining Data. |

During the first six steps of the process, the planning team develops decision performance criteria (i.e., DQOs) that are used to develop the data collection design. The final step of the process involves developing data collection design based on DQOs.

### 3.1 Problem Statement

This plan was developed at the request of the Volpe Center to determine if the former Vermiculite Intermountain facility in Salt Lake City, Utah (now an identified Libby Sister Site) has been impacted by asbestos. Materials present at the site, including soil, dust, and waste/product may contain LA. Also, these materials could potentially produce airborne asbestos within and around the site. Both the bulk materials and any airborne asbestos fibers could present a hazard to anyone located in and surrounding the area of the former processing plant.

The stakeholders associated with decisions made for this site include the Volpe Center, U.S. EPA Region VIII, current owner of the site, Utah Department of Environmental Quality (UDEQ), and any other regulatory agency that addresses health and safety standards for asbestos.

Any affirmative response to the above problem statements presented above will generate alternative actions to address the decision. The following alternative actions could be initiated:

- (a) Initiate removal or remedial action (i.e., cover exposed areas) of the wastes/product at the site.

- (b) Conduct further investigation to determine if the asbestos concentrations are associated with the Libby vermiculite ore (i.e., alternative analyses).
- (c) Take no action.

### 3.2 Identify the Decision

Data collected during this assessment will be used to determine if this particular Libby Sister Site has been impacted by LA asbestos. Specifically, the data will be used to answer the following questions:

- 1) Can any of the wastes/products or soils be considered LA asbestos-containing materials and dust. What are their concentrations?
- 2) How widespread is the contamination?

The above alternative decisions could be modified depending on the LA concentration (i.e., asbestos toxicity) determined at the site.

### 3.3 Inputs to the Decision

The purpose of this step is to identify the information that needs to be obtained and the measurements that need to be taken to resolve the decision statements.

According to the National Emission Standards for Hazardous Air Pollutants (NESHAP) regulations (EPA 1990), a friable asbestos-containing material is defined as "any material containing more than 1 percent asbestos as determined using polarized light microscopy (PLM), that, when dry, can be crumbled, pulverized or reduced to powder by hand pressure." Therefore, to answer the first question, the decision-makers need to know the concentrations of asbestos in the soil and waste/product to determine if they are an asbestos-containing material (ACM) using methods described in Section 5.0 and detailed in Appendix D. The decision-makers also need to know the concentration of asbestos in the breathing air surrounding any personnel coming in contact with ACM or disturbing the environs. This will allow them to determine if those concentrations are above regulatory or other risk-based levels that may pose a threat to human health.

The second question can be answered by evaluating the spatial distribution and concentration of the soil samples. These results will state whether or not asbestos was identified, what type (i.e., LA or chrysotile), and how widespread soil contamination is with respect to the proposed grid or transect locations. Positive microvac samples from surrounding properties will help determine if airborne contamination has migrated offsite.

EPA toxicologists and risk managers will evaluate the asbestos levels and determine whether or not a health threat exists on or off site.

### 3.4 Boundaries for the Removal Assessment

This step defines the spatial and temporal boundaries for the assessment.

#### *Spatial Boundaries*

Concerning the soil and waste/product sampling, the horizontal boundaries for the assessment are approximately the property boundaries (until access agreements are in place for offsite sampling). The vertical boundaries for laboratory analytical sampling are from approximately 1.5 feet below the exposed solid surface to the top of the highest pile or mound of soil. Soil borings will advance to depths below suspected fill as determined by visual inspection. Unless vermiculite is observed in the subgrade fill, sampling will not begin until a depth equivalent to the elevation of the adjacent SLC2 gravel lot is reached. Vertical boundaries can be extended downward based on visual determination to describe the depth of vermiculite or product contamination. For microvac samples, the spatial boundaries for the dust sampling include the interior (including the ceiling) of any onsite building or any equipment planned to be moved offsite. If any ambient air samples are taken, the horizontal boundaries are the property boundaries associated with this particular Libby Sister Site and the vertical boundaries are from the ground surface to approximately six feet above ground (breathing zone). For the personal air monitoring, the boundary is the breathing zone of the affected person.

#### *Temporal Boundaries*

Temporal boundaries include the time frame from when the former site ceased operation (stopped processing vermiculite) through the time of sampling.

### 3.5 Decision Rule

The purpose of this step is to define the parameter of interest, specify the action level (if known), and integrate previous DQO outputs into a single statement/statements that describes a logical basis for determining whether the site has been impacted by asbestos. The parameters of interest are the concentrations of asbestos in soil, waste/product, and air (including the presence or absence of asbestos in the dust). While the primary form of asbestos at the Libby site is the tremolite-actinolite solution series, the combined concentration of all forms of asbestos may be used for decision-making. Site specific action levels for soil and waste/product will be determined by EPA toxicologists and risk managers only if concentrations are considered a potential health threat at this site.

### 3.6 Specify Tolerable Limits on Decision Errors

The purpose of this step is to specify the decision-maker's acceptable limits on decision errors. Decision-makers are interested in knowing the true value of the asbestos concentrations. There are several reasons why decision-makers may not know the true asbestos concentration in soil and waste/product:

- 1) There may be a high degree of variability of asbestos concentration within a sample. Although a sample may be thoroughly mixed, only a small portion of the sample is used for the analysis. This could either result in an under- or over-estimate of the actual asbestos concentration.
- 2) Other fibers with optical properties similar to asbestos minerals may give false positive interferences. This could result in an over-estimate of the actual asbestos concentration.
- 3) The optical properties of asbestos may be obscured by a coating on the fibers. This could result in an under-estimate of the actual asbestos concentration.
- 4) Fibers finer than the resolving power of the microscope (about 0.3  $\mu\text{m}$ ) will not be detected. This could result in an under-estimate of the actual asbestos concentration.
- 5) Heat and acid may alter the index of refraction of asbestos and change its color. This could result in an under-estimate of the actual asbestos concentration.

The null hypothesis for the assessment is that the soils and waste/product have LA asbestos concentrations less than 1 percent or greater and the LA asbestos concentrations in the air and dust are above the regulatory levels.

A false positive or "Type I" decision error refers to the type of error made when the null hypothesis is rejected when it is actually true and a false negative or "Type II" decision error refers to the type of error made when the null hypothesis is accepted when it is actually false. For this assessment, a Type I decision error would result in deciding that soil, waste/product, or air contained asbestos that are below the action levels (i.e., "clean") when they actually did not. A Type II decision error would result in deciding that soil, waste/product, or air contains LA asbestos above the action level (i.e., "dirty") when they actually did not. The closer the reported concentration is to the action level, the higher the probability that an incorrect decision will be made and, therefore, a "gray region" may be established that surrounds the action level. However, for this project, no "gray regions" have been established.

The PLM method for soil and waste/product is semi-quantitative and lacks the necessary precision to establish a "gray region." Therefore, given the lack of quantified analytical precision at the action level, a tolerable decision limit for soil and waste/product analyses of +100% of the action level is reasonable to allow the decision-makers to exercise professional judgement and limit Type I errors. A Type II (low bias) error rate of 100 percent less than the action level would mean that a zero percent result would still lie within the allowable error range. By having a decision error limit of  $\pm 100$  percent, this allows the decision-maker the option to either have the sample reanalyzed, analyzed by another method (e.g., transmission electron microscopy [TEM]), or determine that a site has been impacted based on professional judgment.

For air samples, the "worst case" air samples will be used to determine if a site has been impacted by asbestos. Therefore, a "gray region does not need to be established. However, because human health and safety are involved, a decision error limit below the action level of -50 percent of the action level for air is established. By having a decision error limit of -50 percent for air samples, this allows the decision-makers the option to either have the sample further analyzed (e.g., counting more grids), reanalyzed, analyzed by another method, (e.g., phase contrast microscopy [PCM]), or determine that a site has been impacted (i.e., pose a possible health hazard) based on professional judgment.

### **3.7 Optimize the Decision for Obtaining Data**

The purpose of this step is to identify the most resource-effective sampling design that generates data that satisfy the DQOs in the previous steps. The sampling program described in this SAP is consistent with the DQOs and project objectives for the assessment. However, if during the period of sample collection and/or evaluation, it becomes apparent that the quantity and/or distribution of samples is not sufficient for obtaining the data required to properly characterize soil, waste/product, or air for this assessment, the number, distribution, or methods may be modified to reflect the specific needs of the project. Any changes to this SAP will be approved by EPA and the Volpe Center prior to implementation. In addition, any deviations to this SAP will be noted in the applicable field logbook and subsequently discussed in data summary reports.

## **Section 4**

# **Field Activities and Sampling Procedures**

CDM was tasked by the Volpe Center to provide all personnel, material, equipment, and supplies to complete the tasks identified below related to sampling and investigative support at the SLC2 site. This section describes the procedures that will be followed for information gathering, sample collection, handling, shipping, analysis, and documentation.

### **4.1 Site Information and Access Agreements**

As part of the onsite investigation activities, CDM will gather and verify current and historical information at the SLC2 site (if not previously done). An interview or meetings with the current site contact (property owner) may have already been accomplished or arranged for by the EPA or the Volpe Center prior to arrival onsite. Signed access agreements and insurance documents (if applicable) will be required before any sampling activities commence.

### **4.2 Site Investigation and Photographic Documentation**

CDM will take detailed notes and digital photographs during the investigation and will document the existence of any suspect asbestos materials. Differential global positioning system (GPS) locations and photographs will be taken and logged for sample points in accordance with CDM Standard Operating Procedure (SOP) 4-2 Photographic Documentation of Field Activities.

### **4.3 Soil Sampling**

#### **4.3.1 Selecting Soil Sampling Locations**

Soil samples will be collected from unpaved areas outside of the buildings, on and off site. Additional sampling may include direct-push technology (DPT) methods to multiple depths, including possible coring through asphalt or concrete.

Approximately 70 surface and subsurface soil samples are currently proposed (Figure 4-1) as part of the expanded RA. Actual locations and depths of sample locations are dependent on the needs and goals of the EPA OSC and Volpe Center field team member. Locations will be determined in the field and will be dependent on observed conditions. Grab samples were previously collected for the first walk-through sampling effort (CDM 2003a); however, five-point composite sampling has been recommended for further surface soil investigation of this site. Sampling efforts may change at the discretion of the EPA OSC. The type and location of samples that are collected will be documented on the field sample data sheets (FSDS) (Appendix A).

Approximately 10 subsurface soil borings will be installed outside the perimeter of the former processing building (Figure 4-1). The soil borings will be advanced to at least 1.5 feet below the estimated original ground surface (existing at the time of plant

operations) using DPT methods. Collection of these samples will be dependent on access to the properties.

Approximately two composite soil samples will be collected for PCB analyses. Discrete locations for these samples will be biased towards locations around the base of existing or previously known electrical transformers.

Any additional sampling procedures or changes to the plan (e.g., concrete coring) will be documented in detail in the applicable field logbook.

### 4.3.2 Sample Identification

Each soil sample will be labeled with two unique codes indicating an index identification and location identification. The first code is taken from a list of unique alpha-numeric sequence prepared by CDM for the Region VIII Libby Sister Sites. This coding system is designed to prevent accidental duplication of sample identification numbers and ensures that all samples have a unique identification number assigned to them. These codes start at 1R8-xxxx, with the "1R8" corresponding to the soil sampling team (CDM) and the last five numbers are sequentially numbered so that thousands of unique codes are available. To ensure that the laboratory is "blind" and does not receive certain specific information about a sample, only the index identification code, along with sample date and time, will be used to label sample containers.

The second sample code is a field identification code used by CDM to provide each soil sample with a unique identification code that will allow for the tracking and retrieval of information concerning each sample. Each surface soil sample will be identified by a site identifier, a location identifier, a media identifier, a station identifier, and the depth range of sample collection in inches.

An example is LSS-UTSL-SO-S01-00-02 which indicates that a sample was collected by CDM as part of the Libby Sister Sites asbestos investigation (LSS), that it was collected from the former facility in Salt Lake City, UT (UTSL), that it was a soil sample (SO), from grid station 01 (G01), and that it was collected from a depth of 0 to 2 inches (00-02). The station identifier may also be a feature such as a railroad (R##) or a traverse (T##). The first letter of the location identifier will be changed to a D for duplicate samples (e.g., DTSL). This coding system may be modified to suit field conditions and any modifications will be clearly described in the applicable field logbook.

### 4.3.3 Collecting Soil Samples

All soil samples will be prepared in accordance with the CDM Close Support Facility Soil Preparation Plan (CDM 2003b) and analyzed by National Institute of Occupational Safety and Health (NIOSH) Method 9002, Asbestos (bulk) by PLM Method 9002 (Appendix D). All soil samples will be collected in accordance with CDM Technical SOP 1-3 Surface Soil Sampling and SOP 1-4 Subsurface Soil Sampling (Appendix B), with modifications. The following modifications to SOP 1-3 and SOP 1-4 have been reviewed and approved.

Section 2.2, Discussion - Sample depth for surface soil will generally be 0 to 2 inches from the current ground surface. However, if a sample is required from a compacted dirt road, the depth from 0 to 1 inch will be acceptable assuming a sufficient amount of soil can be obtained. Limited subsurface soil samples may also be required; however, depths will likely be limited to 1.5 feet. Composite samples will be composed of nearly equal portions of soil from five randomly discrete locations within a horizontal radius of approximately 25 feet. The field composite sample will be obtained from an aliquot of total volume of homogenized soil. The actual composite sample for PLM analysis will be prepared at the CDM laboratory in Denver. The laboratory sample will be a split of the processed (i.e., dried, crushed, and homogenized) volume of soil. If vermiculite is observed within the 25 foot radius, it will be included as at least one discrete biased portion of the field sample. Generally, grid and/or traverse segment size will be measured on 50-foot centers.

Section 4.0, Required Equipment - Neither ice bags nor blue ice will be used. Powder-free nitrile gloves will be used for sample collection. No pans, trays, or bowls are necessary, since samples will be placed directly into zipper-top bags. Since the sampling is for asbestos, rather than metals or organic compounds, the use of stainless steel or Teflon-lined sampling instruments is determined not to be necessary. The sampling device may be a garden bulb planter, trowel, DPT macrocore, or other similar sampling device. A list of equipment that may be used for sampling is included in Table 4-1.

Section 5.2.3, Method for Collecting Samples for Nonvolatile Organic or Inorganic Compound Analysis - One-gallon zipper-top bags will be used as sample containers. The one-gallon bags will be filled at least half full. Sampling information will be written directly on the bags using a permanent marker. Sampling instruments do not need to be constructed of stainless steel or Teflon lined. Trays and bowls will not be used, as samples will be placed directly into zipper-top bags. Field homogenization will be performed by manipulating the sampled material inside the zipper-locked bag. All samples will be double bagged for shipping to the Denver lab and further processing.

#### **4.3.4 Sample Documentation**

Sampling activities during this assessment will be documented in the applicable field logbooks (and on FSDSs, Appendix A) to be maintained by the field team in accordance with CDM SOP 4-1 Field Logbook Content and Control (Appendix B). The field team leader will be responsible for maintenance and document control of the field logbook.

#### **4.3.5 Sample Custody, Packaging, and Shipping**

This section details the sample custody and the classifying, identifying, labeling, packaging, and transporting of soil samples collected during this investigation. Procedures will be in accordance with CDM SOPs 1-2 Sample Custody and 2-8 Packaging and Shipping of Environmental Samples (Appendix B) as described below.



Sample classification is necessary to ensure the protection of personnel involved in the shipment of samples, and to maintain the integrity of each sample. Samples obtained at uncontrolled hazardous waste sites are classified as either environmental or hazardous samples. All samples collected during this investigation will be classified as environmental.

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, chain-of-custody (COC) records will be used. The COC record is employed as physical evidence of sample custody and control, and provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. COC procedures will follow the requirements set forth in CDM SOP 1-2 Sample Custody. The following modifications to SOP 1-2 have been reviewed and approved:

Section 5.2, Sample Labels and Tags - Rather than using labels or tags, samples will be identified by writing sample index information directly on the one-gallon zipper-top bags using permanent markers.

Samples collected during this investigation will be packaged and shipped in accordance with CDM SOP 2-8 Packaging and Shipping of Environmental Samples (Appendix B), with modification. The proposed modifications to SOP 2-8 are as follows:

Section 4.0, Required Equipment - No vermiculite or other absorbent material will be used. No bubble wrap or ice will be used.

Section 5.0, Procedures - Lining the cooler with a garbage bag is determined not to be necessary since the samples will already be double-bagged. No vermiculite or other absorbent material will be used to pack the samples. No ice will be used.

### 4.3.6 Quality Control Samples

Quality control (QC) data are necessary to determine precision and accuracy of sample collection techniques and to demonstrate the absence of interference and/or cross-contamination. For this investigation, a soil QC sample will consist of a duplicate taken from an environmental sample in the field following homogenization in the Zipper-top bag.

Soil duplicate samples will be analyzed at a rate of one per twenty soil samples per site (i.e., 5 percent). For each group of twenty sequentially collected natural samples (e.g., 1R8-0001 through 1R8-0010), any one of the twenty samples may be duplicated. The duplicate sample will receive a unique index identification code.

Field duplicate samples may be collected in the field where one portion of this sample (split) will be given to a stakeholder representative. These split samples are collected the same as a duplicate sample using a unique index identification code. However, the remainder of the sample will be archived at Camp Dresser & McKee Inc. (CDM

Inc.'s) laboratory located in Denver, Colorado. A COC form is completed without identifying any analyses and should identify the sample was split as noted in the comment section. The samples, signed copy of the COC, and corresponding field data sheet will be transferred to the stakeholder representative.

No other soil QC samples (e.g., field blanks, interlaboratory splits, etc.) are planned. Rinsate samples are used to evaluate the effectiveness of decontamination procedures. The soil analyses used for this project have a relatively high limit of detection and cross-contamination from sampling equipment would have to be extreme to be detectable in a sample. Decontamination of equipment to be visually clean will be sufficient to avoid cross-contamination and, therefore, no rinsate blanks will be collected.

#### **4.3.7 Equipment Decontamination**

Equipment used to collect, handle, or measure soil samples will be decontaminated in accordance with CDM SOP 4-5 Field Equipment Decontamination at Nonradioactive Sites, with modification (Appendix B). The following modifications to SOP 4-5 have been reviewed and approved:

Section 5.0, Procedures - Decontamination water will not be captured and will be discharged to the ground at the site.

Section 5.3, Sampling Equipment Decontamination - ASTM Type II deionized (DI) water will not be used. Rather, locally available DI water will be used. Decontamination water will be discharged to the ground at the site.

Section 5.6, Waste Disposal - Decontamination water will not be captured and will not be packaged, labeled, or stored as investigation-derived waste.

The decontamination procedure for non-disposable equipment consists of a tap water and alconox wash with brush scrubbing, followed by a tap water rinse, and final DI water rinse. The equipment will then be allowed to air-dry before being wrapped in clean plastic or aluminum foil. All equipment will be decontaminated before coming into contact with any sample. Rinse water will be discharged to the ground at the site. Any deviations from the decontamination procedures will be recorded in the appropriate field logbook.

#### **4.3.8 Health and Safety**

All sampling will be performed in accordance with applicable EPA, Occupational Safety and Health Administration (OSHA), corporate, and site health and safety requirements. CDM has prepared a Site Health and Safety Plan (SHSP) for the site that is attached as Appendix C.

## **4.4 Waste / Product Sampling**

### **4.4.1 Selecting Sample Locations**

Waste/product sampling is not currently scoped for this site. If new sources of product are discovered, then additional sampling locations may be opportunistic. Approximately two waste/product samples will be collected (if found) at the project site. Potential locations would be from around the foundation of the structure used for processing or containing product and the other from outside where the product was stockpiled.

The specifics of any waste/product sampling locations will be determined on-site. The EPA OSC and/or Volpe Center Field Team Member working with the sampling team will determine the number, locations of waste/product samples to be collected at this site and the analytical method. The EPA OSC will also direct the CDM team on the required depth and composite nature of each sample.

### **4.4.2 Sample Identification**

Each bulk sample will be identified with a unique index identification code. The index identification code is a sequential list of sample numbers (IR8-XXXX) that will be used for all of the samples collected by the soil team including soil and bulk waste/product samples. This coding system (see Section 4.3.2) is designed to prevent accidental duplication of sample identification numbers and ensures that all samples have a unique identification number assigned to them. To ensure that the laboratory is "blind" and does not receive certain specific information about a sample, only the index identification code, along with sample date and time, will be used to label sample containers.

Each waste/product sample will also be identified by a site identifier, a location identifier, a media identifier, a station identifier, and the depth range of sample collection in inches. An example is LSS-UTSL-WP-P01-00-06 which indicates that a sample was collected by CDM as part of the Libby Sister Sites asbestos investigation (LSS), that it was collected from the former facility in Salt Lake City, UT (UTSL), that it was a waste/product (WP), that it was from pile 01 (P01), and that it was collected from a depth of 0 to 6 inches (00-06). The station identifier may also be a structure such as a building or shed (B##). The first letter of the location identifier will be changed to a D for duplicate samples (i.e., DTSL). This coding system may be modified to suit field conditions and any modifications will be clearly described in the applicable field logbook.

### **4.4.3 Collecting Samples**

All waste/product samples will be prepared in accordance with the CDM Close Support Facility Soil Preparation Plan (CDM 2003b) and analyzed in accordance with National Institute of Occupational Safety and Health (NIOSH) Method 9002, Asbestos (bulk) by PLM (Appendix D).

The samples will be collected by placing product or waste material into a one gallon plastic zipper-top bag until it is approximately half full. This bag will then be placed into a second plastic zipper-top bag. All waste/product samples will be double bagged. Sampling personnel will wear disposable nitrile gloves while sampling. A new pair of gloves will be donned prior to each sample being collected. Sampling personnel will also wear an appropriate level respiratory protection at all times while collecting waste/product samples.

#### **4.4.4 Sample Documentation**

Sampling activities during this assessment will be documented in the applicable field logbooks and on FSDSs (Appendix A) to be maintained by the field team in accordance with CDM SOP 4-1 Field Logbook Content and Control (Appendix B). The field team leader will be responsible for maintenance and document control of field logbooks.

#### **4.4.5 Sample Custody, Packaging, and Shipping**

This section details the sample custody and the classifying, identifying, labeling, packaging, and transporting of waste/product samples collected during this investigation. Procedures will be conducted in accordance with CDM SOPs 1-2, 2-8, and 4-5 (Appendix B) as described below.

Sample classification is necessary to ensure the protection of personnel involved in the shipment of samples, and to maintain the integrity of each sample. Samples obtained at uncontrolled hazardous waste sites are classified as either environmental or hazardous samples. All samples collected during this investigation will be classified as environmental.

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, COC records will be used. The COC record will be employed as physical evidence of sample custody and control, and provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. COC procedures will follow the requirements set forth in CDM SOP 1-2 Sample Custody. The following modifications to SOP 1-2 have been reviewed and approved:

Section 5.2, Sample Labels and Tags - Rather than using labels or tags, samples will be identified by writing sample information directly on the one-gallon zipper-top bags using permanent markers. All samples will be double-bagged.

Samples collected during this investigation will be packaged and shipped in accordance with CDM SOP 2-8 Packaging and Shipping of Environmental Samples (Appendix B), with modification. The following modifications to SOP 2-8 have been reviewed and approved.

Section 4.0, Required Equipment - No vermiculite or other absorbent material will be used. No bubble wrap or ice will be used.

Section 5.0, Procedures - Lining the cooler with a garbage bag is determined not to be necessary since the samples will already be double-bagged. Procedures related to the packaging of bottles do not apply. No vermiculite or other absorbent material will be used to pack the samples. No ice will be used.

#### 4.4.6 Quality Control Samples

Quality control data are necessary to determine precision and accuracy of sample collection techniques and to demonstrate the absence of interference and/or cross-contamination. For this investigation, a waste/product QC sample will consist of a duplicate taken from an environmental sample in the field following homogenization in the zipper-top bag.

Waste/product duplicate samples will be analyzed at a rate of one per twenty waste/product samples per site (i.e., 5 percent). For each group of twenty sequentially collected natural samples (e.g., 1R8-0020 through 1R8-0040), any one of the twenty samples may be duplicated. The sample will receive a unique index identification code as described in Section 4.4.2.

Split samples may be collected when waste/product samples are collected on property owned (or once owned) by Vermiculite Intermountain (Table 1-1). Split samples may be collected for 100 percent of samples collected on these properties. A split sample will be collected in the same manner as a duplicate sample using a unique index identification code. However, the sample will not be sent to the Denver laboratory for processing and subsequent analysis. A COC form will be completed without identifying any analyses or laboratory. The COC will identify the sample that was split as noted in the comment section. The samples, signed COC, and corresponding field data sheets will be transferred to the stakeholder's representative. A copy of the signed COC will be retained for the project records.

No other waste/product QC samples (e.g., field blanks, interlaboratory splits, etc.) are planned. Rinsate samples are used to evaluate the effectiveness of decontamination procedures. The soil analyses used for this project have a relatively high limit of detection and cross-contamination from sampling equipment would have to be extreme to be detectable in a sample. Decontamination of equipment to be visually clean is sufficient to avoid cross-contamination and, therefore, no rinsate blanks will be collected.

#### 4.4.7 Equipment Decontamination

Equipment used to collect, handle, or measure waste/product samples will be decontaminated in accordance with CDM SOP 4-5 Field Equipment Decontamination at Nonradioactive Sites, with modification (Appendix B). The following modifications to SOP 4-5 have been reviewed and approved:

Section 5.0, Procedures - Decontamination water will not be captured and will be discharged to the ground at the site.

Section 5.3, Sampling Equipment Decontamination - ASTM Type II DI water will not be used. Rather, locally available DI water will be used. Decontamination water will be discharged to the ground at the site.

Section 5.6, Waste Disposal - Decontamination water will not be captured and will not be packaged, labeled, or stored as investigation-derived waste.

The decontamination procedure for non-disposable equipment will consist of a tap water andalconox wash with brush scrubbing, followed by a tap water rinse, and final DI water rinse. The equipment will then allowed to air-dry before being wrapped in clean plastic or aluminum foil. All equipment will be decontaminated before coming into contact with any sample. Rinse water will be discharged to the ground at the site. Any deviations from the decontamination procedures will be recorded in the appropriate field logbook.

#### **4.4.8 Health and Safety**

All sampling will be performed in accordance with all applicable EPA, OSHA, corporate, and site health and safety requirements. CDM has prepared a SHSP for the site that is attached as Appendix C.

### **4.5 Microvacuum Dust Sampling**

#### **4.5.1 Selecting Sample Locations**

Air sampling may be recommended at the site. Microvacuum (dust sampling) locations will be determined based on the size and number of buildings on the project site, current and historic uses of the buildings, and current and historic site conditions. In the case of multiple story buildings or larger buildings, it may be necessary to collect additional microvacuum dust samples to get a more representative sample of the buildings.

The specifics of the dust sampling locations will be determined on site. The Volpe Center Field Team Member working with the sampling team will determine the number and location of microvacuum dust samples to be collected at this site.

#### **4.5.2 Sample Identification**

Each dust sample will be identified with a unique index identification code. The index identification code is a sequential list of sample numbers (2R8-XXXX) that will be used for all of the samples collected by the air team including air, dust and personal air samples. This coding system is designed to prevent accidental duplication of sample identification numbers and ensures that all samples have a unique identification number assigned to them. To ensure that the laboratory is "blind" and does not receive certain specific information about a sample, only the index identification code, along with sample date and time, will be used to label sample cassettes.

Each dust sample will also be identified by a site identifier, a location identifier, a media identifier, and a station identifier, and a sequential number indicating the number of sample from that building.

An example is LSS-UTSL-DU-B01-3-00 which indicates that a sample was collected by CDM as part of the Libby Sister Sites asbestos investigation (LSS), that it was collected from the former facility in Salt Lake City, UT (UTSL), that it was a dust sample (DU), from building 01 (B01), that it was the third sample from that building (3), and space filler to keep the number of characters in the sample code consistent (00). The first letter of the location identifier will be changed to an F for field blanks. This coding system may be modified to suit field conditions and any modifications will be clearly described in the applicable field logbook.

### 4.5.3 Collecting Samples

Microvacuum dust samples will be collected by drawing air through a MCE filter (0.45  $\mu\text{m}$  pore size) at a flow rate of 2.0 L/min for a minimum sampling time of two minutes or until all visible dust or particulate matter has been removed from the sampling area, whichever comes first. The details of the method are provided in ASTM Standard D-5755-95, Microvacuum Sampling and Indirect Analysis Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations (Appendix D). For the purposes of this project there will be one modification to the ASTM Method. The following modification to ASTM Method D-5755-95 is noted:

Section 8.7, Sample Area - The ASTM method indicates that a 100  $\text{cm}^2$  sampling area be vacuumed per cassette. In order to obtain a more representative dust sample from several areas within each building, MACTEC will vacuum three separate 100  $\text{cm}^2$  sampling areas per sampling cassette. Therefore each cassette will represent the dust from a 300  $\text{cm}^2$  area.

### 4.5.4 Sample Documentation

Sampling activities during this assessment will be documented in the applicable field logbooks and on FSDSs (Appendix A) to be maintained by the field team in accordance with CDM SOP 4-1 Field Logbook Content and Control (Appendix B). The field team leader will be responsible for maintenance and document control of field logbooks.

### 4.5.5 Sample Custody, Packaging, and Shipping

This section details the sample custody and the classifying, identifying, labeling, packaging, and transporting of dust samples collected during this investigation.

Sample classification is necessary to ensure the protection of personnel involved in the shipment of samples, and to maintain the integrity of each sample. Dust samples collected during this assessment will be classified as environmental samples.

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, COC records will be used. The COC record will be employed as physical evidence of sample custody and control, and provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. COC procedures will follow the requirements set forth in CDM SOP 1-2 Sample Custody, with modifications (Appendix B). The following modifications to SOP 1-2 have been reviewed and approved:

Section 5.2, Sample Labels and Tags - A label will be affixed to each air sampling cassette prior to being shipped to the appropriate laboratory. This number will correspond to the number assigned to that particular sample in the field data sheets.

Samples collected during this investigation will be packaged and shipped in accordance with CDM SOP 2-8, Packaging and Shipping of Environmental Samples (Appendix B) and ASTM Standard D-5755-97 (Appendix D), with modification. The following modifications to SOP 2-8 are as follows:

Section 4.0, Required Equipment - No vermiculite or other absorbent material will be used. No bubble wrap or ice will be used.

#### **4.5.6 Quality Control Samples**

Quality control methods include both a field and laboratory component. Normally, field personnel will prepare two types of QC samples: duplicates and blanks. However, field duplicates will not be collected for microvacuum samples. In accordance with the ASTM standard, a microvacuum sample must be collected for two minutes or until all visible dust or particulate has been removed from a specified area. Therefore, it may be impossible to duplicate the sampling of dust.

##### *Field Blanks*

The field team will prepare blank samples for dust by labeling unused filter cassettes and submitting them for analysis.

#### **4.5.7 Equipment Decontamination**

This project requires the decontamination of all microvacuum sampling equipment (e.g., pumps, cassette, tubing, etc) prior to sampling and prior to leaving the site.

Equipment used to collect, handle, or measure dust samples will be decontaminated in accordance with CDM SOP 4-5 Field Equipment Decontamination at Nonradioactive Sites, with modification (Appendix B). The following modifications to SOP 4-5 have been reviewed and approved:

Section 5.0, Procedures - Decontamination water will not be captured and will be discharged to the ground at the site.



Section 5.3, Sampling Equipment Decontamination - ASTM Type II DI water will not be used. Rather, locally available DI water will be used. Decontamination water will be discharged to the ground at the site.

Section 5.6, Waste Disposal - Decontamination water will not be captured and will not be packaged, labeled, or stored as investigation-derived waste.

The decontamination procedure for non-disposable equipment will consist of a tap water and alconox wash with brush scrubbing, followed by a tap water rinse, and final DI water rinse. The equipment will then be allowed to air-dry before being wrapped in clean plastic or aluminum foil. All equipment will be decontaminated before coming into contact with any sample. Rinse water will be discharged to the ground at the site. Any deviations from the decontamination procedures will be recorded in the appropriate field logbook.

#### **4.5.8 Health and Safety**

All sampling will be performed in accordance with all applicable EPA, OSHA, corporate, and site health and safety requirements. CDM has prepared a SHSP for the project site that is attached as Appendix C.

### **4.6 Ambient/Personal Air**

#### **4.6.1 Selecting Sample Locations**

If ambient air sampling is conducted, locations will be determined based on the size and number of buildings on the project site, current and historic uses of the buildings, and current and historic site conditions. Ambient air sampling will be performed to determine the asbestos in air concentrations within the buildings.

The EPA OSC and Volpe Center Field Team Member working with the sampling team will determine the number and locations if ambient air samples are to be collected at this site.

Personal air samples will be conducted each day of sampling. The personal air sample will be collected from the breathing zone of the sampler and DPT operator.

#### **4.6.2 Sample Identification**

Each air sample will be identified with a unique index identification code. The index identification code is a sequential list of sample numbers that will be used for all of the samples collected including ambient and personal air samples. This coding system is designed to prevent accidental duplication of sample identification numbers and ensures that all samples have a unique identification number assigned to them. To ensure that the laboratory is "blind" and does not receive certain specific information about a sample, only the index identification code, along with sample date and time, will be used to label sample cassettes.

Each air sample will also be identified by a site identifier, a location identifier, a media identifier, a station identifier, and the height from ground surface of sample collection, in inches.

An example is LSS-UTSL-AA-B02-2-72 which indicates that a sample was collected by CDM as part of the Libby Sister Sites asbestos investigation (LSS), that it was collected from the former facility in Salt Lake City, UT (UTSL), that it was an ambient air sample (AA), from building 02 (B02), second sample from that building (2), and that it was collected from 72 inches above ground surface (72). The first letter of the location identifier will be changed to an F for field blanks. This coding system may be modified to suit field conditions and any modifications will be clearly described in the applicable field logbook. Personal air samples will be indicated by the alpha characters (PA).

### 4.6.3 Collecting Samples

Air samples will be collected by drawing air through a MCE filter (0.45  $\mu\text{m}$  pore size) at a specified flow rate for a specified period of time. The details of the method are provided in EPA SOP 2015 Asbestos Sampling (Appendix D). Under normal circumstances, ambient air samples will be collected at a flow rate of 10 L/min over a 6- to 7-hour sampling period. This results in a total sampling volume 4200 liters.

Depending on the sampling conditions, work activities, the level of asbestos in the air, and the level of interfering particles in the air, the flow rate, total sampling time, and/or sampling volume may require modifications. The decision to modify the flow rate, time, or volume will be made by the Volpe Center Field Team Member working with the sampling team.

### 4.6.4 Sample Documentation

Sampling activities during this removal assessment will be documented in the applicable field logbooks (and on FSDSs, see Appendix A) to be maintained by the field team in accordance with CDM SOP 4-1 Field Logbook Content and Control (Appendix B). The field team leader will be responsible for maintenance and document control of field logbooks.

### 4.6.5 Sample Custody, Packaging, and Shipping

This section details the sample custody and the classifying, identifying, labeling, packaging, and transporting of air samples collected during this investigation.

Sample classification is necessary to ensure the protection of personnel involved in the shipment of samples, and to maintain the integrity of each sample. Air samples collected during this assessment will be classified as environmental samples.

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, COC records will be used. The COC record will be

employed as physical evidence of sample custody and control, and provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. COC procedures will follow the requirements set forth in CDM SOP 1-2 Sample Custody, with modifications (Appendix B). The following modifications to SOP 1-2 have been reviewed and approved:

Section 5.2, Sample Labels and Tags - A label will be affixed to each air sampling cassette prior to being shipped to the appropriate laboratory. This number will correspond to the number assigned to that particular sample in the field data sheets.

Samples collected during this investigation will be packaged and shipped in accordance with CDM SOP 2-8 Packaging and Shipping of Environmental Samples (Appendix B) and ASTM Standard D-5755-97 (Appendix B), with modification. The following modifications to SOP 2-8 have been reviewed and approved:

Section 4.0, Required Equipment - No vermiculite or other absorbent material will be used. No bubble wrap or ice will be used.

#### **4.6.6 Quality Control Samples**

The field team will prepare one type of QC sample: field blanks.

##### *Field Blanks*

The field team will prepare blank samples for air by labeling unused filter cassettes and submitting them for analysis.

#### **4.6.7 Equipment Decontamination**

This project requires the decontamination of all air sampling equipment (e.g., pumps, cassette, tubing, etc) prior to sampling and prior to leaving the site.

Equipment used to collect, handle, or measure air samples will be decontaminated in accordance with CDM SOP 4-5 Field Equipment Decontamination at Nonradioactive Sites, with modification (Appendix B). The following modifications to SOP 4-5 have been reviewed and approved:

Section 5.0, Procedures - Decontamination water will not be captured and will be discharged to the ground at the site.

Section 5.6, Waste Disposal - Decontamination water will not be captured and will not be packaged, labeled, or stored as investigation-derived waste.

The decontamination procedure for non-disposable equipment consists of a tap water andalconox wash with brush scrubbing, followed by a tap water rinse, and final DI water rinse. The equipment will then be allowed to air-dry before being wrapped in clean plastic or aluminum foil. All equipment will be decontaminated before coming into contact with any sample. Rinse water will be discharged to the ground at the site.

Any deviations from the decontamination procedures will be recorded in the appropriate field logbook.

#### **4.6.8 Health and Safety**

All sampling will be performed in accordance with applicable EPA, OSHA, corporate, and site health and safety requirements. CDM has prepared a SHSP that is specific to this project attached as Appendix C.

## Section 5

# Laboratory Analytical Methods

All soil and waste/product samples will be sent to the following location for sample preparation:

CDM Inc. Laboratory  
2710 Walnut Street  
Denver, Colorado 80202  
Attn: Todd Burgesser  
(303)295-3935

All soil and waste/product samples will be processed in accordance with the CDM Close Support Facility Soil Preparation Plan (CDM 2003b) (Appendix D). Following preparation, all soil and waste/product will be analyzed by PLM/NIOSH 9002 (Appendix D). Removal decisions will be based on the fine ground sample portion analytical result.

Any air and dust samples will be sent directly to the analytical laboratory and will not require any preliminary processing at the CDM Inc. Laboratory. Analytical services for soil, waste/product, dust, and air samples will be conducted by one of the following laboratories:

EMSL Analytical Inc.  
107 Haddon Avenue  
Westmont, NJ 08108  
Attn: Mr. Robert DeMalo  
(800) 220-3675 ext. 1256

Reservoir Environmental Services Inc.  
1827 Grant Street  
Denver, CO 80203  
Attn: Ms. Jeanne Orr  
(303) 830-1986

The most appropriate analytical methods for each environmental medium will depend on the type and level of asbestos contamination and on the detection levels needed to assess hazard and/or nature and extent of contamination. Table 5-1 identifies the analytical methods that will be utilized during the assessment. Analytical methods are included as Appendix D.

The laboratory used for all sample analysis will be accredited under the Laboratory Accreditation Program as sponsored by the American Industrial Hygiene Association (AIHA). The laboratory will also actively participate in the NIOSH Proficiency Analytical Testing Program for Laboratory Quality Control for asbestos. Lastly, the laboratory will be fully accredited for TEM and PLM analysis under the National

Voluntary Laboratory Accreditation Program as sponsored by the National Institute of Standards and Technology (NIST).

Soil analyses for PCBs will be conducted directly on field samples without any preprocessing by CDM Inc. The method used for analysis is EPA 8082/8081A. The following laboratory will be used for all analyses other than asbestos:

Alpha Analytical Laboratories  
Eight Walkup Drive  
Westborough, MA 01581-1019

## **Section 6**

# **Quality Assurance / Quality Control**

Because the results of the sampling covered by this SAP will be used for decisions made during focused preliminary assessments and are not intended for future decisions regarding remedial actions or risk, a moderate level of QA/QC is warranted.

Field QA/QC requirements are identified in Section 4.0. Laboratory QA/QC requirements are identified in the general laboratory quality assurance plan maintained by the selected laboratory.

### **6.1 Instrument Calibration and Frequency**

No field measurements will be made; therefore, no calibration of field equipment will be necessary.

Laboratory instrumentation, used for sample analyses, will be calibrated in accordance with USEPA or NIOSH methodologies. Calibrations must be acceptable before any measurements on investigative samples are conducted. Traceable calibration standards are obtained by the analytical laboratories. All documentation relating to receipt, preparation, and use of standards will be recorded in the appropriate laboratory logbooks. This information will be forwarded as part of the analytical data package as described in Section 7.0.

### **6.2 Assessment and Response Actions**

The following sections describe activities for assessing the effectiveness of the implementation of the project and associated QA/QC. The purpose of the appraisal is to ensure that the SAP is implemented as prescribed. At this time, no audits or self-assessments are scheduled for this project; however, in case they do occur, their elements are described in the following sections.

#### **6.2.1 Audits / Self-Assessments**

Evaluation of office and field activities and laboratory analyses may be conducted through oversight of analytical procedures through project audits or self-assessments. Project self-assessments are reviews of projects or examination of project activities conducted by technical personnel who are knowledgeable in the project-specific requirements, whether the requirements involve office, field, or laboratory work.

Audits/self assessments are conducted to ensure that the technical requirements of the projects are being met. Office audits/self assessments are conducted to ensure that document control, and other QA requirements are being met. The purpose of the field project audit/self assessments is to document field sampling and analysis procedures, to determine if activities are proceeding in accord with project

requirements, and to document any changes, additions, or deletions that have occurred during field sampling and analysis and to provide rapid feedback to the project staff and to facilitate corrective action and continuous improvement.

Laboratory audits/self assessments evaluate laboratory procedures to ensure that they follow Good Laboratory Practices (GLP) Guidelines and to ensure that they do not conflict with project requirements. If conflicts are noted, these must be addressed so that project requirements are met.

Other possible audits/self assessments that may be carried out over the course of the project including:

- Review and verification of procedures followed as part of real-time control charting of QC samples analyzed via field and contract laboratory procedures
- Evaluation of the flow of electronic data
- Review and verification of hardcopy data

Audits/self assessments may review the data flow, verify data entry procedures, and evaluate whether data management QC protocols will be observed. If audits/self assessments resulting from review of any of the procedures reveal that project requirements are not met, then an improvement plan (Figure 6-1) or corrective action plan for the deficiency must be requested, reviewed, and reported. Results for all audits or self-assessments will be submitted to the corporate QA director identified on the signature page of this document. Information in the reports includes:

- Type of project audit/self assessment (field, office, laboratory, data management, etc.)
- Date of audit/self assessment
- Summary of situation or procedures reviewed
- Results of the audit/self assessment and plan of action describing any non-conformances noted
- Corrective action request(s) (CAR) or improvement plan, if non-conformance noted
- Date by which CAR or improvement plan action must be received with response and any necessary documentation

If a CAR or improvement plan is required, a follow-up verification must be performed within 20 working days upon receipt of the CAR or improvement plan response to ensure that corrective actions were implemented. More detailed information regarding corrective action procedures is provided in the next section.



## 6.2.2 Corrective Action Procedures

Two types of corrective actions may result from project audits/self assessments: immediate and long-term. Immediate corrective actions include correcting deficiencies or errors or correcting inadequate procedures. Long-term corrective actions are designed to eliminate the sources of deficiencies or errors. If either type of corrective action is deemed necessary following a project audit/self assessment, each step in the following procedures must be documented:

- Identify the deviation or deficiency
- Request a corrective action
- Report the problem through a CAR or through an improvement plan to the QA director
- Review the corrective action response
- Perform a follow-up verification to ensure the deviation is not recurring

## Section 7

# Data Reporting and Deliverables

Following completion of analysis for each sample delivery group (SDG), the laboratory will prepare a report that will include a tabulation of all sample results, COC forms, and laboratory QA/QC analyses pertinent to that SDG. The laboratory will fax or deliver the report to the appropriate personnel (CDM for soil and waste/product or MACTEC for dust and air). The laboratory will also submit an electronic copy of the data results to CDM. The results of the sampling will be maintained using EQUIS.

Final laboratory reports will be provided to CDM that include all sample results, necessary narratives, replicate analyses, continuing calibration results (if available), and any other QC results associated with the analyses.

Following completion of all field activities and receipt of all final SDGs, CDM will prepare a draft RA report (RA) for the former Vermiculite Intermountain site. The draft RA will include a brief description of the field program, laboratory test results, maps showing the locations and concentrations of the samples collected, assessment of the DQOs, and any deviations from the SAP. Copies of the laboratory results will be attached as an appendix. Three copies of each draft RA will be submitted to the Volpe Center task order contracting officer's technical representative (TO COTR) for review and comment:

Project Manager  
US/DOT/RSPA/Volpe Center  
Attn: John McGuiggin, PE  
55 Broadway, Kendall Square  
Cambridge, MA 02142  
Ph: (617)494-2574  
Fax: (617)494-2789  
Cell: (617)320-4164  
Email: [mcguiggin@volpe.dot.gov](mailto:mcguiggin@volpe.dot.gov)

Any comments received will be addressed and five copies of the final RA will be issued to the TO COTR.

## Section 8

### References

ACGIH 1998, 1998 TLVs® and BEIs®, American Conference of Governmental Industrial Hygienists, Inc., Publication 0098.

CDM 2001. Draft letter to Mr. John McGuiggin dated May 11, 2001 re: Libby Sister Site Walk-through at Former W.R. Grace Facility, Salt Lake City, UT on March 27, 2001.

CDM 2003a. Letter Report Summarizing Sampling Activities at Former Vermiculite Intermountain Facility - SLC2. Revision 1, January.

CDM. 2003b. Close Support Facility Soil Preparation Plan, Libby Asbestos Site, Operable Unit 4. April 25

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EPA 1990. Asbestos/NESHAP Regulated Asbestos Containing Materials Guidance. EPA 340/1-90-018. December.

EPA 2000. Guidance for the Data Quality Objectives Process. EPA QA/G4. EPA/600/R-96/055. August.

IRIS 1999, IRIS On-line Database, Asbestos - CASRN 1332-21-4, Last Revised -- 09/26/1988.

NIOSH 1999, Pocket Guide to Chemical Hazards, National Institute for Occupational Safety and Health, Publication 99-115.

OSHA 1998a, Asbestos Standard for Industry, Occupational Safety and Health Administration Rules Codified at 29 CFR 1910.1001 et seq.

OSHA 1998b, Asbestos Standard for Construction, Occupational Safety and Health Administration Rules Codified at 29 CFR 1926.1101 et seq.

USDA 1977. Soil Survey of Lincoln County Area. U.S. Department of Agriculture National Resources Conservation Service Soil Survey Staff.

Volpe Center 2000, Time-Critical Removal Action, Screening plant (Operable Unit 02), EPA Libby Asbestos Project, Libby, Montana. Prepared by Camp, Dresser and McKee, Inc. August.

## Figures

### Figure 6-1 Improvement Plan

<b>CDM FEDERAL PROGRAMS CORPORATION IMPROVEMENT PLAN</b>	
Project No./Title: _____	
Client/Contract: _____	
Project Manager: _____	QA Coordinator: _____
Situation (Attach Additional Pages as Required):	
Situation Identified By: _____	Date: _____
Plan of Action (Attach Additional Pages as Required):	
Responsible for Action: _____	
Scheduled Completion Date: _____	
Actual Completion Date: _____	
Project Manager Signature: _____	Date: _____

## Tables

**Table 3-1 Summary of Available PCM and TEM Based Exposure Levels for Asbestos**

Agency	Description	Required Analysis	Nominal Value	Reference
ACGIH	TLV-TWA	PCM	0.1 f/cc (0.1 f/ml)	American Conference of Governmental Industrial Hygienists, Inc. ACGIH, 1998
NIOSH	REL 100 minute TWA in a 400L sample (all forms)	PCM	0.1 f/cc (0.1 f/ml)	National Institute for Occupational Safety and Health NIOSH, 1999
OSHA	PEL (TWA) all forms	PCM	0.1 f/cc (0.1 f/ml)	Asbestos Standard for Industry, Occupational Safety and Health Administration Rules OSHA, 1998 29 CFR 1910.1001
OSHA	PEL (ceiling) 30 minute average (all forms)	PCM	1.0 f/cc (1.0 f/ml)	Asbestos Standard for Construction, Occupational Safety and Health Administration Rules OSHA, 1998 29 CFR 1926.1101
EPA (AHERA)	Level to determine the completion of a response action in Schools	TEM	70 structures per square millimeter (s/mm <sup>2</sup> )	Asbestos Hazard Emergency Response Act of 1986 EPA, 1987 40 CFR 763
EPA (IRIS)	Inhalation unit risk	PCM	0.23 f/cc (f/ml)	On-line Database, Asbestos IRIS, 1999
EPA (EMSL)	Measure of Work Site Cleanliness	PCM	Less than or equal to 0.01 f/cc (0.01 f/ml)	EMSL, 1985

**Table 4-1 Supply Checklist**

Note: This supply and equipment list should be used in addition to the list found in the specific SOPs.

**General**

SAP  
SOPs  
HASP  
Access agreement  
Sample labels/tags/pens  
Permanent markers  
Field book  
Pin flags  
Digital camera  
Garbage bags  
100-foot tape measure  
GPS unit  
Cellular phone  
File box  
Color pencils  
Express shipping labels  
Field forms (COCs and Data Sheets)  
Tool kit  
1-gallon zipper-top bags

**Equipment Decontamination/Personal**

**Protective Equipment**

Rubber overboots  
Tyvek coveralls  
Liquid soap  
Disposable gloves  
Respirators w/cartridges (see HASP)  
Duct tape  
respirator cleaning kit  
5-gallon water-boy  
paper towels  
safety glasses  
eye wash kit  
first aid kit  
tap water  
garden sprayer  
long-handle brush  
aluminum foil  
tubs for decontamination

**Soil and Waste/Product**

bulb planting tool, trowel, or other  
sampling

site maps  
wood stakes  
300-ft measuring tape  
flagging  
plastic sheeting

**Air and Dust Sampling**

high-volume sample pumps (2-12L/min)  
low-volume sample pumps  
tygon tubing  
sample stands  
air-flow calibrator  
tubing/cassett adaptors  
shrink-wrap  
extension cord  
50 filter cassettes (0.45 um, MCE filter)  
air sampling forms  
filter cassettes  
tyvek  
metric ruler  
filter paper  
8 1/2 x 11 plastic sheets  
masking tape  
tape measure  
flash light w/batteries  
ear plugs  
magnifying glass  
disposable hand cleaners  
100 cm<sup>2</sup>-template



**Table 5-1 Summary of Analytical Methods**

<b>Matrix</b>	<b>Analysis</b>	<b>Holding Time</b>	<b>Analytical Method</b>
Soil*	Preparation Asbestos (bulk) by PLM	6 months	See CDM 2003b NIOSH Method 9002
Waste/ Product	Preparation Asbestos (bulk) by PLM	6 months	See CDM 2003b NIOSH Method 9002
Dust	International Standard, Determination of asbestos fibers	6 months	ISO 10312
Air personal	Asbestos and Other Fibers by PCM and TEM (AHERA)	6 months	NIOSH 7400 EPA 40 CFR Part 763 Final Rule

\*Two composite soil samples will be analyzed for PCBs by EPA Method 8092/8081A.

**Appendix A**

**Field Sample Data Sheets**

# LIBBY SISTER SITE FIELD SAMPLE DATA SHEET

## STATIONARY AIR

Scenario No.: \_\_\_\_\_ Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_ Sampling Date: \_\_\_\_\_

Address: \_\_\_\_\_ Owner: \_\_\_\_\_

Land Use: Residential School Commercial Mining Roadway Other ( )

Sampling Team: PES CDM Other \_\_\_\_\_ Names: \_\_\_\_\_

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID			
Location ID			
Sample Group			
Location Description			
Category (circle)	FS _____ Blank _____ Rep _____	FS _____ Blank _____ Rep _____	FS _____ Blank _____ Rep _____
Matrix Type (circle)	Indoor _____ Outdoor _____ NA _____	Indoor _____ Outdoor _____ NA _____	Indoor _____ Outdoor _____ NA _____
Filter Diameter (circle)	27mm _____ 37mm _____	27mm _____ 37mm _____	27mm _____ 37mm _____
Pore Size (circle)	TEM- .45 _____ PCM- .08 _____	TEM- .45 _____ PCM- .08 _____	TEM- .45 _____ PCM- .08 _____
Flow Meter Type (circle)	Rotometer _____ DryCal _____	Rotometer _____ DryCal _____	Rotometer _____ DryCal _____
Pump ID Number			
Flow Meter ID No.			
Start Date			
Start Time			
Start Flow (L/min)			
Stop Date			
Stop Time			
Stop Flow (L/min)			
Pump fault? (circle)	No _____ Yes _____	No _____ Yes _____	No _____ Yes _____
MET Station onsite?	No _____ Yes _____	No _____ Yes _____	No _____ Yes _____
Pre/Post (circle)	Pre _____ Post _____ Clear _____ NA _____	Pre _____ Post _____ Clear _____ NA _____	Pre _____ Post _____ Clear _____ NA _____
Field Comments			
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

**LIBBY SISTER SITE FIELD SAMPLE DATA SHEET****DUST**

Scenario No.: \_\_\_\_\_ Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_ Sampling Date: \_\_\_\_\_

Address: \_\_\_\_\_ Owner: \_\_\_\_\_

Land Use: Residential School Commercial Mining Roadway Other ( )

Sampling Team: PES CDM Other \_\_\_\_\_ Names: \_\_\_\_\_

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID			
Location ID			
Sample Group			
Location Description			
Category (circle)	FS Blank	FS Blank	FS Blank
Matrix Type (circle)	Building, Vehicle, NA Other	Building, Vehicle, NA Other	Building, Vehicle, NA Other
Sample Area (cm <sup>2</sup> )	300	300	300
Filter Diameter	25mm	25mm	25mm
Pore Size (circle)	TEM- .45 PCM- .08	TEM- .45 PCM- .08	TEM- .45 PCM- .08
Flow Meter Type (circle)	Rotometer Dry-Cal	Rotometer Dry-Cal	Rotometer Dry-Cal
Pump ID Number			
Flow Meter ID No.			
Start Date			
Start Time			
Start Flow (L/min)			
Stop Date			
Stop Time			
Stop Flow (L/min)			
Pump fault? (circle)	No Yes	No Yes	No Yes
Field Comments	100 cm <sup>2</sup>  100 cm <sup>2</sup>  100 cm <sup>2</sup>	100 cm <sup>2</sup>  100 cm <sup>2</sup>  100 cm <sup>2</sup>	100 cm <sup>2</sup>  100 cm <sup>2</sup>  100 cm <sup>2</sup>
	Entered ___ Validated ___	Entered ___ Validated ___	Entered ___ Validated ___

**LIBBY SISTER SITE FIELD SAMPLE DATA SHEET****PERSONAL AIR**

Scenario No.: \_\_\_\_\_ Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_ Sampling Date: \_\_\_\_\_

Address: \_\_\_\_\_ Owner: \_\_\_\_\_

Sampling Team: PES CDM Other \_\_\_\_\_ Names: \_\_\_\_\_

Land Use: Residential School Commercial Mining Roadway Other ( )

Person Sampled: \_\_\_\_\_ SSN: \_\_\_\_\_ Task: \_\_\_\_\_

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID			
Location ID			
Sample Group			
Location Description			
Category (circle)	FS Blank Rep _____	FS Blank Rep _____	FS Blank Rep _____
Matrix Type (circle)	Indoor Outdoor NA	Indoor Outdoor NA	Indoor Outdoor NA
Filter Diameter (circle)	25mm 37mm	25mm 37mm	25mm 37mm
Pore Size (circle)	TEM- .45 PCM- .08	TEM- .45 PCM- .08	TEM- .45 PCM- .08
Flow Meter Type (circle)	Rotometer DryCal	Rotometer DryCal	Rotometer DryCal
Pump ID Number			
Flow Meter ID No.			
Start Date			
Start Time			
Start Flow (L/min)			
Stop Date			
Stop Time			
Stop Flow (L/min)			
Pump fault?	No Yes	No Yes	No Yes
MET Station onsite?	No Yes	No Yes	No Yes
Sample Type	TWA EXC NA	TWA EXC NA	TWA EXC NA
Field Comments			
	Entered ___ Validated ___	Entered ___ Validated ___	Entered ___ Validated ___

**LIBBY SISTER SITES FIELD SAMPLE DATA SHEET**  
**SOIL-LIKE MATERIALS**

Scenario No.: \_\_\_\_\_ Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_ Sampling Date: \_\_\_\_\_

Address: \_\_\_\_\_ Owner: \_\_\_\_\_

Land Use: (circle) Residential School Commercial Mining Roadway Other ( )

Sampling Team: (circle) CDM PES Other \_\_\_\_\_ Names: \_\_\_\_\_

Data Item	Sample 1	Sample 2	Sample 3
Index ID			
Location ID			
Sample Group			
Location Description (circle)	Yard Soil Garden Soil Play Area Driveway Other _____	Yard Soil Garden Soil Play Area Driveway Other _____	Yard Soil Garden Soil Play Area Driveway Other _____
Category (circle)	FS FD _____	FS FD _____	FS FD _____
Matrix Type (circle)	Mining Waste Subsurface Soil Surface Soil Fill Other _____	Mining Waste Subsurface Soil Surface Soil Fill Other _____	Mining Waste Subsurface Soil Surface Soil Fill Other _____
Type (circle)	Grab Comp. # subsamples _____	Grab Comp. # subsamples _____	Grab Comp. # subsamples _____
Sample Time			
Top Depth (in.)			
Bottom Depth (in.)			
Map Location			
Field Comments			
	Entered _____ Validated _____	Entered _____ Validated _____	Entered _____ Validated _____

## **Appendix B**

### **Standard Operating Procedures (SOPs)**

## **CDM Technical Standard Operating Procedures**

- SOP 1-2      Sample Custody
- SOP 1-3      Surface Soil Sampling
- SOP 1-4      Subsurface Soil Sampling
- SOP 2-1      Packaging and Shipping of Environmental Samples
- SOP 4-1      Field Logbook Content and Control
- SOP 4-2      Photographic Documentation of Field Activities
- SOP 4-5      Field Equipment Decontamination at Nonradioactive Sites

## **EPA Standard Operating Procedures**

- SOP 2015      Asbestos Sampling

## **ASTM Designation: D 5755-95**

D 5755-95      Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by TEM for Asbestos Structure Number Concentrations



## SAMPLE CUSTODY

SOP 1-2

Revision: 3

Date October 12, 2001

Page 1 of 9

Prepared: David O. Johnson

Technical Review: Jackie Mosher

QA Review: Doug Updike

Approved: [Signature]

Issued: Rosemary Justin 10/12/01

Signature/Date

### 1.0 OBJECTIVE

Due to the evidentiary nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until their derived data are introduced as evidence in legal proceedings. To maintain and document sample possession, sample custody procedures are followed. All paperwork associated with the sample custody procedures will be retained in CDM Federal Programs Corporation (CDM Federal) files unless the client requests that it be transferred to them for use in legal proceedings or at the completion of the contract.

Note: Sample custody documentation requirements vary with the specific EPA region or client. This SOP is intended to present basic sample custody requirements, along with common options. Specific sample custody requirements should be presented in the project-specific quality assurance (QA) project plan or project-specific modification or clarification form (See Section U-1).

### 2.0 BACKGROUND

#### 2.1 Definitions

**Sample** – A sample is material to be analyzed that is contained in single or multiple containers representing a unique sample identification number.

**Sample Custody** – A sample is under custody if:

1. It is in your possession.
2. It is in your view, after being in your possession.
3. It was in your possession and you locked it up.
4. It is in a designated secure area.

**Chain-of-Custody Record** – A chain-of-custody record is a form used to document the transfer of custody of samples from one individual to another.

**Custody Seal** – A custody seal is a tape-like seal that is part of the chain-of-custody process and is used to detect tampering with samples after they have been packed for shipping.

## **SAMPLE CUSTODY**

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**Sample Label** – A sample label is an adhesive label placed on sample containers to designate a sample identification number and other sampling information.

**Sample Tag** – A sample tag is attached with string to a sample container to designate a sample identification number and other sampling information. Tags may be used when it is difficult to physically place adhesive labels on the container (e.g., in the case of small air sampling tubes).

### **3.0 RESPONSIBILITIES**

**Sampler** – The sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

**Field Team Leader (FTL)** – The FTL is responsible for ensuring that strict chain-of-custody procedures are maintained during all sampling events. The FTL is also responsible for coordinating with the subcontractor laboratory to ensure that adequate information is recorded on custody records. The FTL determines whether proper custody procedures were followed during the fieldwork and decides if additional samples are required.

**Field Sample Custodian** – The field sample custodian, when designated by the FTL, is responsible for accepting custody of samples from the sampler(s) and properly packing and shipping the samples to the laboratory assigned to do the analyses. A field sample custodian is typically designated only for large and complex field efforts.

### **4.0 REQUIRED SUPPLIES**

- Chain-of-custody records (applicable client or CDM Federal forms)
- Custody seals
- Sample labels or tags
- Clear tape

### **5.0 PROCEDURES**

#### **5.1 Chain-of-Custody Record**

This procedure establishes a method for maintaining custody of samples through use of a chain-of-custody record. This procedure will be followed for all samples collected or split samples accepted.

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### Field Custody

1. Collect only the number of samples needed to represent the media being sampled. To the extent possible, determine the quantity and types of samples and sample locations prior to the actual fieldwork. As few people as possible should handle samples.
2. Complete sample labels or tags for each sample, using waterproof ink.

### Transfer of Custody and Shipment

1. Complete a chain-of-custody record for all samples (see Figure 1 for an example of a chain-of-custody record. Similar forms may be used when requested by the client). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the sample custodian in the appropriate laboratory.
  - The date/time will be the same for both signatures when custody is transferred directly to another person. When samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures. Common carriers are not required to sign the chain-of-custody record.
  - In all cases, it must be readily apparent that the person who received custody is the same person who relinquished custody to the next custodian.
  - If samples are left unattended or a person refuses to sign, this must be documented and explained on the chain-of-custody record.

NOTE: If a field sample custodian has been designated, he/she may initiate the chain-of-custody record, sign and date as the relinquisher. The individual sampler(s) must sign in the appropriate block, but does (do) not need to sign and date as a relinquisher (refer to Figure 1).

2. Package samples properly for shipment and dispatch to the appropriate laboratory for analysis. Each shipment must be accompanied with a separate chain-of-custody record.
3. Include a chain-of-custody record identifying its content in all shipments (refer to Figure 1). The original record will accompany the shipment, and the copies will be retained by the FTL and, if applicable, distributed to the appropriate sample coordinators. Freight bills will also be retained by the FTL as part of the permanent documentation.

# SAMPLE CUSTODY

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**Figure 1**  
**EXAMPLE CDM Federal Chain-of-Custody Record**

**CDM** Federal Programs Corporation  
A subsidiary of Camp Dresser & McKee Inc.

125 Maiden Lane, 5th Floor  
New York, NY 10038  
(212) 785-9123  
Fax: (212) 785-6114

## CHAIN OF CUSTODY RECORD

PROJECT ID.		FIELD TEAM LEADER		LABORATORY AND ADDRESS				DATE SHIPPED																																																																																																																											
PROJECT NAME/LOCATION				LAB CONTRACT:				AIRBILL NO.																																																																																																																											
<b>MEDIA TYPE</b> 1. Surface Water 2. Groundwater 3. Leachate 4. Field QC 5. Soil/Sediment 6. Oil 7. Waste 8. Other _____		<b>PRESERVATIVES</b> 1. HCl, pH <2 2. HNO <sub>3</sub> , pH <2 3. NaOH, pH >12 4. H <sub>2</sub> SO <sub>4</sub> , pH <2 5. Zinc Acetate, pH >9 6. Ice Only 7. Not Preserved 8. Other _____		<b>SAMPLE TYPE</b> G = Grab C = Composite		<b>ANALYSES (List no. of containers submitted)</b> <table border="1"> <tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr> <tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr> <tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr> <tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr> <tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr> <tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr> <tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr> <tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr> <tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr> <tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr> </table>																																																																																																																													
SAMPLE LOCATION NO.	LABORATORY SAMPLE NUMBER	PRESERVATIVES ADDED	MEDIA TYPE	SAMPLE TYPE	19 DATE	TIME SAMPLED						REMARKS (Note if MS/MSD)																																																																																																																							
1.																																																																																																																																			
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10.																																																																																																																																			

**SAMPLER SIGNATURES:**

RELINQUISHED BY: (print)	DATE/TIME	RECEIVED BY: (print)	DATE/TIME	RELINQUISHED BY: (print)	DATE/TIME	RECEIVED BY: (print)	DATE/TIME
(signature)		(signature)		(signature)		(signature)	
RELINQUISHED BY: (print)	DATE/TIME	RECEIVED BY: (print)	DATE/TIME	RELINQUISHED BY: (print)	DATE/TIME	RECEIVED BY: (print)	DATE/TIME
(signature)		(signature)		(signature)		(signature)	

**COMMENTS:**

DISTRIBUTION: White and yellow copies accompany sample shipment to laboratory; yellow copy retained by laboratory. Pink copy retained by samplers.

1/98

**NOTE:** If requested by the client, different chain-of-custody records may be used. Copies of the template for this record may be obtained from the Fairfax Graphics Department.

## SAMPLE CUSTODY

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### Procedure for Completing CDM Federal Example Chain-of-Custody Record (Refer to Figure 1.)

The following procedure is to be used to fill out the CDM Federal chain-of-custody record. The record is provided herein as an example chain-of-custody record. If another type of custody record (i.e., provided by the EPA contract laboratory program or a subcontract laboratory) is used to track the custody of samples, the custody record should be filled out in its entirety.

1. Record project number.
2. Record FTL for the project (if a field sample custodian has been designated, also record this name in the "Remarks" box).
3. Record the name and address of the laboratory to which samples are being shipped.
4. Enter the project name/location or code number.
5. Record overnight courier's airbill number.
6. Record sample location number.
7. Record sample number.
8. Note preservatives type and reference number.
9. Note media type (matrix) and reference number.
10. Note sample type.
11. Enter date of sample collection.
12. Enter time of sample collection in military time.
13. When required by the client, enter the names or initials of the samplers next to the sample location number of the sample they collected.
14. List parameters for analysis and the number of containers submitted for each analysis.
15. Enter MS/MSD (matrix spike/matrix spike duplicate) if sample is for laboratory quality control or other remarks (e.g. sample depth).
16. Sign the chain-of-custody record(s) in the space provided. All samplers must sign each record.
17. If sample tags are used, record the sample tag number in the "Remarks" column.
18. Record date shipped.
19. The originator checks information entered in Items 1 through 16 and then signs the top left "Relinquished by" box, prints his/her name, and enters the current date and time (military).

## **SAMPLE CUSTODY**

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20. Send the top two copies (usually white and yellow) with the samples to the laboratory; retain the third copy (usually pink) for the project files. Retain additional copies for the project file or distribute as required to the appropriate sample coordinators.
21. The laboratory sample custodian receiving the sample shipment checks the sample label information against the chain-of-custody record. Sample condition is checked and anything unusual is noted under "Remarks" on the chain-of-custody record. The laboratory custodian receiving custody signs in the adjacent "Received by" box and keeps the copy. The white copy is returned to CDM Federal.

### **5.2 Sample Labels and Tags**

Unless the client directs otherwise, sample labels or tags will be used for all samples collected or accepted for CDM Federal projects.

1. Complete one label or tag with the information required by the client for each sample container collected. A typical label or tag would be completed as follows (see Figure 2 for example of sample tag; labels are completed with the equivalent information):
  - Record the project code (i.e., project or task number).
  - Enter the station number (sample number) if applicable.
  - Record the date to indicate the month, day, and year of sample collection.
  - Enter the time (military) of sample collection.
  - Place a check to indicate composite or grab sample.
  - Record the station (sample) location.
  - Sign in the space provided.
  - Place a check next to "yes" or "no" to indicate if a preservative was added.
  - Place a check under "Analyses" next to the parameters for which the sample is to be analyzed. If the desired analysis is not listed, write it in the empty slot. Note: Do not write in the box for "laboratory sample number."
  - Place or write additional relevant information under "Remarks".
2. Place adhesive labels directly on the sample containers. Place clear tape over the label to protect from moisture.
3. Securely attach sample tags to the sample bottle. On 80 oz. amber bottles, the tag string may be looped through the ring style handle and tied. On all other containers, it is recommended that the string be looped around the neck of the bottle, then twisted and re-looped around the neck until the slack in the string is removed.

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Figure 2  
EXAMPLE Sample Tag

Designate		Lab	Comp.	Time	Month/Day/Year	Station No.	Project Code	Station Location	Preservative: Yes <input type="checkbox"/> No <input type="checkbox"/>	
									ANALYSES	
									BOD Antons	
									Solids (res) (res) (ss)	
									COD, TOC, Nutrients	
									Phenolics	
									Mercury	
									Metals	
									Cyanide	
									Oil and Grease	
									Organics GC/MS	
									Priority Pollutants	
									Volatile Organics	
									Pesticides	
									Mutagenicity	
									Bacteriology	
									Remarks:	
									Tag No. Lab Sample No.	
									3-3023215	

NOTE: Equivalent sample labels or tags may be used.

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### **5.3 Custody Seals**

Custody seals must be placed on the shipping containers (e.g., picnic cooler) prior to shipment. The seal should be signed and dated by a field team member.

Custody seals may also be placed on individual sample bottles. Check with the client or refer to EPA regional guidelines for direction.

### **5.4 Sample Shipping**

The CDM Federal standard operating procedure listed below defines the requirements for packaging and shipping environmental samples.

- CDM Federal SOP 2-1, Packaging and Shipping of Environmental Samples

## **6.0 RESTRICTIONS/LIMITATIONS**

Check with the EPA region or client for specific guidelines. If no specific guidelines are identified, this procedure should be followed.

For EPA Contract Laboratory Program (CLP) sampling events, combined chain-of-custody/traffic report forms or other EPA-specific records may be used. Refer to regional guidelines for completing these forms.

The EPA FORMS II Lite™ software may be used to customize sample labels and custody records when directed by the client or the CDM Federal project manager.



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### 7.0 REFERENCES

U.S. Environmental Protection Agency, *EPA Guidance for Quality Assurance Project Plans*, EPA QA/G-5, EPA/600/R-98/018, February 1998, Section B3.

U.S. Environmental Protection Agency, *National Enforcement Investigations Center, Multi-Media Investigation Manual*, EPA-330/9-89-003-R, Revised March 1992, p.85.

U.S. Environmental Protection Agency, *Contract Laboratory Program (CLP), Guidance for Field Samplers*, EPA-540-R-00-003, Draft Final, June 2001, Section 3.2.

U.S. Environmental Protection Agency, *FORMS II Lite™ User's Guide*, March 2001

U.S. Environmental Protection Agency, Region IV, *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, May 1996, Section 3.3.

U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plan*, EM 200-1-3, February 2001, Appendix F.

# SURFACE SOIL SAMPLING

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Prepared: Del Baird

Technical Review: Brian Jerks

QA Review: Matt Brookshire

Approved: [Signature]

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Issued: [Signature]

Signature/Date

## 1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to define the techniques and the requirements for collecting surface soil samples.

## 2.0 BACKGROUND

Surface soils are generally defined as the soils extending from ground surface to approximately 1 foot below ground surface (bgs). Surface soil samples are frequently collected from 0 to 6 inches bgs. The techniques and protocol described herein may be used to collect other surface media, including sediment and sludge.

### 2.1 Definitions

Surface Soil - The soil that exists down from the surface approximately one foot (30 centimeters). Depending on application, the soil interval to be sampled will vary.

Grab Sample - A discrete portion or aliquot taken from a specific location at a given point in time.

Composite - Two or more sub-samples taken from a specific media and site at a specific point in time. The sub-samples are collected and mixed, then a single average sample is taken from the mixture.

Spoon/Scoop - A small stainless steel or Teflon® utensil approximately 6 inches in length with a stem-like handle.

Trowel - A small stainless steel or Teflon® shovel approximately 6 to 8 inches in length with a slight (approximately 140°) curve across. The trowel has a stem-like handle (for hand operation). Samples are collected with a spooning action.

### 2.2 Discussion

Surface soil samples are collected to determine the type(s) and level(s) of contamination and are often important to risk assessment. These samples may be collected as part of an investigative plan, site-specific sampling plan, and/or as a screen for "hot spots," which may require more extensive sampling.

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Sediment(s) and sludge(s) that have been exposed by evaporation, stream rerouting, or any other means are collected by the same methods as those for surface soil(s). Typically, the top 1 to 2 centimeters (cm) of material, including vegetation, are carefully removed before collection of the sample.

Surface soil and exposed sediment or sludge are collected using stainless steel and/or Teflon®-lined trowels or scoops.

### **2.3 Associated Procedures**

- CDM Federal SOP 1-2, Sample Custody
- CDM Federal SOP 2-1, Packaging and Shipping of Environmental Samples
- CDM Federal SOP 4-1, Field Logbook Content and Control
- CDM Federal SOP 4-5, Field Equipment Decontamination at Non-radioactive Sites

### **3.0 RESPONSIBILITIES**

**Site Manager** - The site manager is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any other SOPs pertaining to specific media sampling.

**Field Team Leader** - The field team leader is responsible for ensuring that field personnel collect surface soil samples in accordance with this and other relevant procedures.

### **4.0 REQUIRED EQUIPMENT**

- Insulated cooler and waterproof sealing tape
- Ice bags or "blue ice"
- Latex or appropriate gloves
- Plastic zip-top bags
- Personal protective clothing and equipment
- Stainless steel and/or Teflon®-lined spatulas and pans, trays, or bowls
- Stainless steel and/or Teflon®-lined trowels or spoons (or equipment as specified in the site-specific plans)
- Plastic sheeting
- Project plans (work plan/health and safety plan)
- Appropriate sample containers
- Field logbook
- Indelible ink pen and/or marker
- Sample chain-of-custody forms
- Custody seals
- Decontamination supplies

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Additional equipment is discussed in Section 5.2.2, VOC Field Sampling/Preservation Methods.

### **5.0 PROCEDURES**

#### **5.1 Preparation**

The following steps must be followed when preparing for sample collection:

1. Don the appropriate personal protective clothing as dictated by the site-specific health and safety plan.
2. Locate sampling location(s) in accordance with project documents (e.g., work plan) and document pertinent information in the appropriate field logbook.
3. Processes for verifying depth of sampling must be specified in the site-specific plans.
4. Place clean plastic sheeting on a flat, level surface near the sampling area, if possible, and place equipment to be used on the plastic; place the insulated cooler(s) on separate plastic sheeting. Cover all equipment and supplies with clean plastic sheeting when not in use.
5. A clean, decontaminated trowel, scoop, or spoon will be used for each sample collected. Other equipment may be used (e.g., shovels) if constructed of stainless steel.

#### **5.2 Collection**

The following general steps must be followed when collecting surface soil samples:

1. Surface soil samples are normally collected from the least contaminated to the most-contaminated areas.
2. Document the sampling events, recording the information in the designated field logbook. Document any and all deviations from SOPs in the field logbook and include rationale for changes. See CDM Federal SOP 4-1.
3. Carefully remove stones, vegetation, snow, etc. from the ground surface in the immediate vicinity of the sampling location.
4. First collect required sample aliquot for volatile analyses as well as any other samples that would be degraded by aeration. Follow with collection of samples for other analyses.
5. Decontaminate sampling equipment between locations. See CDM Federal SOP 4-5.

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### 5.2.1 Method for Collecting Samples for Volatile Organic Compound (VOC) Analysis

The requirements for collecting grab samples of surface soil for VOCs or other samples degraded by aeration are as follows:

1. VOC samples shall be collected with the least disturbance possible.
2. VOC samples shall be collected as grab samples; however, the method of collection will vary from site to site, based on data quality objectives and the degree of known or suspected contamination.
3. Complete sample label by filling in the appropriate information and securing the label to the container. Cover the sample label with a piece of clear tape.
4. Use a clean stainless steel or Teflon®-lined trowel or spoon (or tube) to collect sufficient material in one grab to fill the sample containers.
5. With the aid of a clean stainless steel spatula, quickly fill the sample containers directly from the sampling device, removing stones, twigs, grass, etc., from the sample. Fill the containers *as full and compact as possible to minimize headspace*.
6. Immediately secure the Teflon®-lined cap(s) on the sample container(s).
7. Wipe the containers with a clean Kimwipe or paper towel to remove any residual soil from the exterior of the container.
8. Place the containers in individual zip-top plastic bag(s) and seal the bag(s).
9. Pack all samples as required. Include properly completed documentation, and affix signed and dated custody seals to the cooler lid.

**NOTE:** A trip blank should be included with sample coolers containing VOC samples. QA sample requirements vary from project to project. Consult the project-specific work plan for requirements.

### 5.2.2 Field Sampling/Preservation Methods

The following four sections contain SW 846 methods for sampling and field preservation. These methods include EN CORE™ Sampler Method for low-level detection limits, EN CORE™ Sampler Method for high level/detection limits/screening, acid preservation, and methanol preservation. These methods are very detailed and contain equipment requirements at the beginning of each section.

**NOTE:** Some variations from these methods may be required depending on the contracted analytical laboratory, such as sample volume.

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### **5.2.2.1 EN CORE™ SAMPLER COLLECTION FOR LOW LEVEL ANALYSES ( $\geq 1$ UG/KG)**

#### **EN CORE™ Sampling Equipment Requirements**

The following equipment is required for low-level analysis:

- Three 5-g samplers

**NOTE:** The sample volume requirements are general requirements. Actual sample volumes, sizes, and quantities may vary depending on client or laboratory requirements.

- One 4-ounce widemouth glass jar or applicable container for moisture analysis
- One T-handle
- Paper towels

#### **EN CORE™ Sampling Steps for Low Level Analysis**

1. Remove sampler and cap from package and attach T-handle to sampler body.
2. Quickly push the sampler into a freshly exposed surface of soil until the O-ring is visible within the hole on the side of the T-handle. If the O-ring is not visible within this window, then the sampler is not full.
3. Extract the sampler and wipe the sampler head with a paper towel so that the cap can be tightly attached.
4. Push cap on with a twisting motion to secure to the sampler body.
5. Rotate the sampler stem counterclockwise until stem locks in place to retain sample within the sampler body.
6. Fill out sample label and attach to sampler.
7. Repeat procedure for the other two samplers.
8. Collect moisture sample in 4-ounce widemouth jar using a clean stainless steel spoon or trowel.
9. Store samplers at 4° Celsius. Samples must be shipped and delivered to the analytical laboratory for extraction within 48 hours.

**NOTE:** Verify state requirements for extraction/holding times.

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### **5.2.2.2 ACID PRESERVATION SAMPLING FOR LOW LEVEL ANALYSES ( $\leq 1$ UG/KG)**

#### **Acid Preservation Sampling Equipment Requirements**

The following equipment and supplies are required if field acid preservation is required:

- One 40mL VOA vial with acid preservation (for field testing of soil pH)
- Two pre-weighed 40mL VOA vials with acid preservative and stir bar (for lab analysis)
- Two pre-weighed 40mL VOA vials with water and stir bar (in case samples cannot be pre-preserved)
- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a pre-weighed vial that contains methanol (for screening sample and/or high level analysis)
- One 4-oz widemouth glass jar or applicable container for moisture analysis
- One 2-oz jar with acid preservative (in case additional acid is needed due to high soil pH)
- One appropriately sized scoop capable of delivering 1g of solid sodium bisulfate
- pH paper
- Weighing scale capable of reading to 0.01g
- Set of balance weights used in daily balance calibration
- Gloves for working with pre-weighed sample vials
- Paper towels
- Sodium bisulfate acid ( $\text{NaHSO}_4$ )
- A cutoff plastic syringe or other coring device capable of collecting sufficient sample volume (5g)

#### **Testing Effervescing Capacity of Soils**

Soils must be tested with acid to determine the amount of effervescing that will occur when preserved with acid. Effervescing will drive off VOCs as well as create a high pressure in a sealed vial that could result in the explosion of the sample container. The following steps provide information on the effervescing capacity of the soil.

1. Place approximately 5g of soil into a vial that contains acid preservative and no stir bar.
2. Do not cap this vial as it may EXPLODE upon interaction with the soil.
3. Observe the sample for gas formation (due to carbonates in the soil).
4. If vigorous or sustained gas emissions are observed, then acid preservation is not acceptable to preserve the sample.
  - In this case the samples need to be collected in the VOA vials with only water and a stir bar. The vials with acid preservative CANNOT be used.

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5. If a small amount or no gas formation occurs, then acid preservation is acceptable to preserve the sample. Keep this testing vial for use in the buffering test detailed below.

- In this case the samples need to be collected in the VOA vials with the acid preservative and a stir bar.

### **Testing Buffering Capacity of Soils**

The soils must be tested to determine the quantity of acid that is required to achieve a pH reading of  $\leq 2$  standard units (STUs). The following steps will assist in determining this quantity.

1. If acid preservation is acceptable for sampling soils, then the sample vial that was used to test the effervescing capacity of the soils can be used to test the buffering capacity.
2. Cap the vial that contains 5g of soil, acid preservative, and no stir bar from Step 1 in the effervescing test.
3. Shake the vial gently to homogenize the contents.
4. Open the vial and check the pH of the acid solution with pH paper.
  - If the pH paper reads below 2, then the sampling can be done in the two pre-weighed 40mL VOA vials with the acid preservative and stir bar. Since the pH was below 2, it is not necessary to add additional acid to the vials.
  - If the pH paper reads above 2, then additional acid needs to be added to the sample vial.
5. Use the jar with the solid sodium bisulfate acid and add another 1g of acid to the sample.
6. Cap the vial and shake thoroughly again.
7. Repeat Step 4.
  - If the pH paper reads below 2, then the sampling can be done in the two pre-weighed 40mL VOA vials with the acid preservative and stir bar and one extra gram of acid.
  - Make a note of the extra gram of acid needed so the same amount of acid can be added to the vials the lab will analyze.
  - If the pH paper reads above 2, repeat Steps 5 through 7 until the sample pH  $\leq 2$  STUs.

Now that the soil chemistry has been determined, the actual sampling can occur. The procedure stated below assumes the correct vials are used based on the guidance discussed.



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### **Sample Preservation Steps**

1. Wear gloves during all handling of pre-weighed vials.
2. Add more acid if necessary (based on the buffering capacity testing discussed in the previous section).
3. Quickly collect a 5g sample using a cut off plastic syringe or other coring device designed to deliver 5g of soil from a freshly exposed surface of soil.
4. Carefully wipe exterior of sample collection device with a clean paper towel.
5. Quickly transfer the sample to the appropriate VOA vial, use caution when extruding the sample to prevent splashing of the acid in the vial.
6. Remove any soil from the threads of the sample vial using a clean paper towel.
7. Cap vial and weigh the jar to the nearest 0.01g.
8. Record exact weight on sample label.
9. Repeat sampling procedure for the duplicate VOA vial.
10. Weigh the vial containing methanol preservative in it to the nearest 0.01g. If the weight of the vial with methanol varies by more than 0.01g from the original weight recorded on the vial, discard the vial. If the weight is within tolerance, it can be used for soil preservation below.
11. Take the empty jar or the jar that contains the methanol preservative.
12. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size is dependent on who is doing the sampling and requirements specified by the analytical laboratory.
13. Carefully wipe the exterior of the collection device with a clean paper towel.
14. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol, be careful not to splash the methanol outside of the vial.
15. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided, using only one vial of methanol preservative per sample jar.

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16. Remove any soil from the threads of the sample vial using a clean paper towel and cap the jar.
17. Weigh the jar with sample to the nearest 0.01g and record the weight on the sample label.
18. Collect dry weight sample using a clean stainless steel spoon or trowel.
19. Store samples at 4° Celsius.
20. Ship sample containers to the analytical laboratory with plenty of ice in accordance with Department of Transportation (DOT) regulations (CORROSIVE, FLAMMABLE LIQUID, POISON).

### **5.2.2.3 EN CORE™ SAMPLER COLLECTION FOR HIGH LEVEL ANALYSES (≥200 UG/KG)**

#### **EN CORE™ Sampling Equipment Requirements**

The following equipment is required for high-level analysis.

- One 25-g sampler or one 5-g sampler

**NOTE:** The volume requirements specified are general requirements. Actual sample volumes, container sizes, and quantities may vary depending on client or laboratory requirements.

- One 4-oz widemouth glass jar of applicable container specified for moisture analysis
- One T-handle
- Paper towels

#### **EN CORE™ Sampling Steps for High Level Analysis**

1. Remove sample and cap from package and attach T-handle to sampler body.
2. Quickly push the sampler into freshly exposed surface of soil until the O-ring is visible within the hole/window on the side of the T-handle. If the O-ring is not visible within the window/hole, then the sampler is not full.
3. Use a clean paper towel to quickly wipe the sampler head so that the cap can be tightly attached.
4. Push cap on with a twisting motion to secure to the sampler body.
5. Fill out sample label and attach to sampler.

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6. Rotate sampler stem counterclockwise until the stemlocks in place to retain the sample within the sampler body.
7. Collect moisture sample in 4-oz widemouth glass jar or designated container using a clean stainless steel spoon or trowel.
8. Store samplers at 4° Celsius. Samples must be shipped and delivered to the analytical laboratory for extraction within 48 hours.

**NOTE:** Verify state requirements for extraction/holding times.

### **5.2.2.4 METHANOL PRESERVATION SAMPLING FOR HIGH LEVEL ANALYSES ( $\geq 200$ UG/KG)**

#### **Methanol Preservation Sampling Equipment Requirements**

- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a pre-weighed vial that contains methanol (laboratory grade)
- One dry weight cup
- Weighing balance that accurately weighs to 0.01g
- Set of balance weights used in daily balance calibration
- Latex gloves
- Paper towels
- Cutoff plastic syringe or other coring device to deliver 5g or 25g of soil

#### **Sampling Preservation Steps**

1. Wear gloves during all handling of pre-weighed vials.
2. Weigh the vial containing methanol preservative in it to the nearest 0.01g. If the weight of the vial with methanol varies by more than 0.01g from the original weight recorded on the vial, discard the vial. If the weight is within tolerance, it can be used for soil preservation/ collection below.
3. Take the empty jar or the jar that contains the methanol preservative.
4. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil.
5. Carefully wipe the exterior of the collection device with a clean paper towel.

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6. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol, be careful not to splash the methanol outside of the vial. Again, the type of jar used is dependent on who is doing the laboratory analysis.
7. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided, using only one vial of methanol preservative per sample jar.
8. Remove any soil from the exterior of the vial using a clean paper towel and cap the sample jar.
9. Weigh the jar with the soil in it to the nearest 0.01g and record the weight on the sample label.
10. Collect dry weight sample using a clean stainless steel spoon or trowel.
11. Store samples at 4° Celsius.
12. Ship sample containers with plenty of ice to the analytical laboratory in accordance with DOT regulations (CORROSIVE. FLAMMABLE LIQUID. POISON).

### **5.2.3 Method for Collecting Samples for Nonvolatile Organic or Inorganic Compound Analysis**

The requirements for collecting samples of surface soil for nonvolatile organic or inorganic analyses are as follows:

1. Label each sample container with the appropriate information. Secure the label by covering it with a piece of clear tape.
2. Use a decontaminated stainless steel or Teflon®-lined trowel or spoon to obtain sufficient sample from the required interval and sub-sampling points, if necessary, to fill the specified sample containers.
3. Empty the contents of each fill of the sampling device directly into a clean stainless steel or Teflon®-lined tray or bowl.
4. Homogenize the sample by mixing with a spoon, spatula, or trowel.
5. Use the spoon, spatula, or trowel to distribute the uniform mixture into the labeled sample containers. Fill organic sample containers first, then inorganics.
6. Secure the appropriate cap on each container immediately after filling it.
7. Wipe the sample containers with a clean Kimwipe or paper towel to remove any residual soil.

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8. Place sample containers in individual zip-top plastic bags and seal the bags.
9. Pack all samples as required. Include properly completed documentation, and affix custody seals to the cooler lid.
10. Decontaminate sampling equipment according to CDM Federal SOP 4-5.

### 6.0 RESTRICTIONS/LIMITATIONS

When grab sampling for VOC analysis or for analysis of any other compound(s) that may be degraded by aeration, it is necessary to minimize sample disturbance and, hence, analyze loss. The representativeness of this sample is difficult to determine because the collected sample represents a single point, is not homogenized, and has been disturbed.

### 7.0 REFERENCES

U.S. Department of Energy, Hazardous Waste Remedial Actions Program, *Quality Control Requirements For Field Methods*, DOE/HWP-69/R1, July 1990 or current revision.

U.S. Department of Energy, Hazardous Waste Remedial Actions Program, *Standard Operating Procedures For Site Characterizations*, DOE/HWP-100/R2, September 1996 or current revision.

U.S. Environmental Protection Agency, *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987 or current revision.

U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste*, Physical/Chemical Methods (SW-846), Third Edition, November 1986, (as amended by Update III, June 1997). Method 5035: Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples.

# SUBSURFACE SOIL SAMPLING

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Revision: 4

Date: June 20, 2001

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Prepared: Del Baird

Technical Review: Brian Jenks

QA Review: Matt Brookshire

Approved: [Signature]

Signature/Date

Issued: [Signature]

Signature/Date

## 1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to define the techniques and requirements for collecting soil samples from the unconsolidated zone. Techniques include use of hand augers, Shelby tubes, continuous core samplers, and split-spoon samplers.

## 2.0 BACKGROUND

### 2.1 Definitions

Unconsolidated Zone - The layer of soil above bedrock that exists in a relatively loose state.

Hand Auger - A stainless steel cylinder (bucket) approximately 3 to 4 inches in diameter and 1 foot in length, open at both ends with the bottom edge designed to twist into the soil and cut out a soil core. The bucket collects the soil sample. The auger has a T-shaped handle (for hand operation) attached to the top of the bucket by extendable stainless steel rod(s).

Shelby Tube - A cylindrical sampling device, generally made of steel, which is driven into the subsurface soil through the hollow-stem auger. The tube, once retrieved, may be capped and the undisturbed soil sample extruded in the laboratory prior to analysis.

Split-Spoon - A cylindrical sampling device, generally made of carbon steel, which fits into a hollow stem auger. The spoon is hinged lengthwise, which allows the sample to be retrieved by opening ("splitting") the spoon.

Subsurface Soil - The soil which exists deeper than approximately 1 foot (30 centimeters) from the surface but above bedrock or any other consolidated material.

Grab Sample - A discrete portion or aliquot taken from a specific location at a given point in time.

Liner - A cylindrical sampling device, generally made of brass, stainless steel, or Teflon® that is placed inside a split-spoon or hand auger bucket to collect undisturbed samples.

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**Composite Sample** - Two or more sub-samples taken from a specific media and site at a specific point in time. The sub-samples are collected and mixed, then a single average sample is taken from the mixture.

**Auger Flight** - A steel section length attached to an auger to extend the auger as coring depth increases.

### 2.2 Discussion

Shallow subsurface soil samples (to depths between 6 inches and 10 feet) may be collected using hand augers. However, soil samples collected with a hand auger are commonly of poorer quality than those collected by split-spoon or Shelby tube samplers since the soil sample is disturbed in the augering process. Split-spoon and Shelby tube liners may be used to prevent loss of volatile organic compounds (VOCs). The size and construction material of sampling devices should be selected based on project and analytical objectives and defined in site-specific plans.

### 2.3 Associated Procedures

- CDM Federal SOP 1-2, Sample Custody
- CDM Federal SOP 2-1, Packaging and Shipping of Environmental Samples
- CDM Federal SOP 3-5, Lithologic Logging
- CDM Federal SOP 4-1, Field Logbook Content and Control
- CDM Federal SOP 4-5, Field Equipment Decontamination at Nonradioactive Sites

### 3.0 RESPONSIBILITIES

**Site Manager** - The site manager is responsible for ensuring that field personnel are trained in the use of this procedure and the required equipment, and for ensuring that subsurface soil samples are collected in accordance with this procedure and any other SOPs pertaining to specific media sampling.

**Field Team Leader** - The field team leader is responsible for ensuring that field personnel collect subsurface soil samples in accordance with this SOP and other relevant procedures.

### 4.0 REQUIRED EQUIPMENT

#### 4.1 General

- Site-specific plans
- Field logbook
- Indelible black ink pens and markers
- Labels and appropriate forms/documentation for sample shipment
- Clear, waterproof tape
- Appropriate sample containers

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- Insulated cooler(s) and waterproof sealing tape
- Ice bags or "blue ice"
- Latex or appropriate gloves
- Plastic zip-top bags
- Personal protective clothing and equipment
- Stainless steel and/or Teflon®-lined spatulas and pans, trays, or bowls
- Plastic sheeting

Additional equipment is discussed in Section 5.2.2 VOC Field Sampling/Preservation Methods.

### **4.2 Manual (Hand) Augering**

- T-handle
- Hand auger: flighted-, bucket-, or tube-type auger as required by the site-specific plans
- Extension rods
- Wrench(es), pliers

### **4.3 Split-Spoon and Shelby Tube Sampling**

- Drill rig equipped with a 140-lb drop hammer and sufficient hollow-stem augers to drill to the depths required by the site-specific plans.
- Sufficient numbers of split-spoon or Shelby tube samplers so that at least one is always decontaminated and available for sampling. Three split-spoon or Shelby tube samplers are generally the minimum necessary. (Shelby tubes are usually used only once.)
- Split-spoon liners (as appropriate).
- Wrench(es), hammer.

## **5.0 PROCEDURES**

### **5.1 Preparation**

1. Don the appropriate personal protective clothing as dictated by the site-specific health and safety plan.
2. Locate sampling location(s) in accordance with project documents (e.g., work plan) and document pertinent information in the appropriate field logbook. When possible, reference locations back to existing site features such as buildings, roads, intersections, etc..
3. Processes for verifying depth of sampling must be specified in the site-specific plans.



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4. Clear away vegetation and debris from the surface at the boring location.
5. Prepare an area next to the sample collection location for laying out cuttings by placing plastic sheeting on the ground to cover the immediate area surrounding the borehole.
6. Set up a decontamination line, if decontamination is required in accordance with CDM Federal SOP 4-5.

### 5.2 Collection

The following general steps must be followed when collecting all subsurface soil samples:

1. VOC samples or samples degraded by aeration shall be collected first and with the least disturbance possible. These samples shall be collected as grab samples.
2. Sampling information shall be recorded in the field logbook and on any associated forms. Describe lithology, according to CDM Federal SOP 3-5, in the field logbook or on the lithologic log form.
3. Specific sampling devices to be used shall be identified in the site-specific plans and recorded in the field logbook.
4. Care must be taken to prevent cross-contamination and misidentification of samples.
5. Processes for verifying depth of sampling must be specified in the site-specific plans.
6. Sample bottles for VOC analysis should be filled completely to minimize headspace.

#### 5.2.1 Manual (Hand) Augering

The following steps must be followed when collecting hand-augered samples:

1. Auger to the depth required for sampling. Place cuttings on plastic sheeting or as specified in the site-specific plans. If possible, lay out the cuttings in stratigraphic order.
2. Throughout the augering, make detailed notes concerning the geologic features of the soil or sediments in the field logbook.
3. Cease augering when the top of the specified sampling depth has been reached. If required, remove the auger from the hole and decontaminate the auger or use a fresh auger. Then obtain the sample.
4. Collect a grab sample for VOC analyses (or samples that may be degraded by aeration)

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immediately and place in sample container. Sample bottles should be filled completely to minimize headspace.

5. Label the sample container with the appropriate information. Secure the label by covering it with a piece of clear tape.
6. Remaining sample should be homogenized for other analyses prior to placing samples in the appropriate containers. Label containers as required.
7. Wipe containers with a clean Kimwipe or paper towel to remove residual soil from the exterior of the container(s).
8. Place the containers in zip-top plastic bags and seal the bags. Pack samples in a cooler with ice.
9. Proceed with further sampling, as required by the site-specific plans.
10. When all sampling is complete, dispose of cuttings, plastic sheeting, etc., as specified in the site-specific plans.
11. Complete the field logbook entry and other appropriate forms, being sure to record all relevant information before leaving the site.
12. Properly package all samples for shipment and complete all necessary sample shipment documentation. Remand custody of samples to the appropriate personnel. See CDM Federal SOPs 1-2 and 2-1 or site-specific plans.

### 5.2.2 Field Sampling/Preservation Methods

The following four sections contain SW 846 Methods for sampling and field preservation. These methods include ENCORE™ Sampler Method for low-level detection limits, ENCORE™ Sampler Method for high-level limits/screening, acid preservation, and methanol preservation. These methods may be used if required by the EPA Region, client, or governing sample plan. These methods are very detailed and contain equipment requirements at the beginning of each section.

Note: Some variations from these methods may be required depending on the contracted analytical laboratory, such as sample volume.

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### 5.2.2.1 EN CORE™ SAMPLER COLLECTION FOR LOW LEVEL ANALYSES ( $\geq 1$ UG/KG)

#### EN CORE™ Sampling Equipment Requirements

The following equipment is required for low level analysis:

- Three 5g samplers

**NOTE:** The sample volume requirements specified are general requirements. Actual sample volume and/or container sizes, may vary depending on client or laboratory requirements.

- One dry weight cup
- One T-handle
- Paper towels

#### EN CORE™ Sampling Steps for Low Level Analysis

1. Remove sampler and cap from package and attach T-handle to sampler body.
2. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.
3. Extract sampler and wipe the sampler head with a paper towel so that the cap can be tightly attached.
4. Push cap on with a twisting motion to secure.
5. Fill out sample label and attach to sampler.
6. Repeat procedure for the other two samplers.
7. Collect dry weight sample (60 ml).
8. Store samplers at 4 degrees (°) Celsius.

Ship sample containers with plenty of ice to the laboratory within 40 hours of collection.

**NOTE:** Verify state requirements for extraction/holding times.

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### 5.2.2.2 ACID PRESERVATION SAMPLING FOR LOW LEVEL ANALYSES ( $\geq 1$ UG/KG)

#### Acid Preservation Sampling Equipment Requirements

The following equipment and supplies are required if field preservation is required:

- One 40mL VOA vial with acid preservation (for field testing of soil pH). Two pre-weighed 40mL VOA vials with acid preservative and stir bar (for lab analysis).
- Two pre-weighed 40mL VOA vials with water and stir bar (in case sample effervesces).
- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a pre-weighed vial that contains methanol (for screening sample and/or high level analysis).
- One dry weight cup.
- One 2oz jar with acid preservative (in case additional acid is needed due to high soil pH).
- One scoop capable to deliver about 1g of solid sodium bisulfate.
- pH paper.
- Weighing balance that weighs to 0.01g (with an accuracy  $\pm 0.1$ g).
- Set of balance weights used in daily balance calibration.
- Gloves for working with pre-weighed sample vials.
- Paper towels.
- Sodium bisulfate acid ( $\text{NaHSO}_4$ ) acid.
- A cutoff plastic syringe or other coring device to deliver 5g or 25g of soil.

#### Testing Effervescing Capacity of Soils

Soils must be tested with acid to determine the amount of effervescing that will occur when preserved with acid. Effervescing will drive off VOCs as well as create a very high pressure in a sealed vial which could explode. The following steps will provide information on the effervescing capacity of the soil.

1. Place approximately 5g of soil into a vial that contains acid preservative and no stir bar.
2. Do not cap this vial as it may EXPLODE upon interaction with the soil.
3. Observe the sample for gas evolution (due to carbonates in the soil).
4. If vigorous or sustained gas evolution occurs, then acid preservation is not acceptable to preserve the sample.
  - In this case the samples need to be collected in the VOA vials with only water and a stir bar. The vials with acid preservative CANNOT be used.
5. If a small amount or no gas evolution occurs, then acid preservation is acceptable to preserve the sample. Keep this testing vial for use in the buffering test detailed below.

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- In this case the samples need to be collected in the VOA vials with the acid preservative and a stir bar.

### Testing Buffering Capacity of Soils

The soils must be tested to determine the quantity of acid that is required to reach a less than 2 pH reading. The following steps will assist in determining this quantity.

1. If acid preservation is acceptable for sampling soils, then the sample vial that was used in the effervescing testing can be used here for the buffering testing.
2. Cap the vial that contains approximately 5g of soil, acid preservative, and no stir bar from Step 1 in the effervescing testing.
3. Shake the vial gently to attempt to make a homogenous solution.
4. When done, open the vial and check the pH of the acid solution with pH paper.
  - If the pH paper reads below 2, then the sampling can be done in the two pre-weighed 40mL VOA vials with the acid preservative and stir bar. Since the pH was below 2, it is not necessary to add additional acid to the vials.
  - If the pH paper reads above 2, then additional acid needs to be added to the sample vial.
5. Use the jar with the solid sodium bisulfate acid and add another 1g of acid to the sample.
6. Cap the vial and shake thoroughly again.
7. When done, open the vial and check the pH of the acid solution with a new piece of pH paper.
  - If the pH paper reads below 2 then the sampling can be done in the two pre-weighed 40mL VOA vials with the acid preservative and stir bar and one extra gram of acid.
  - Make a note of the extra gram of acid needed so the same amount of acid can be added to the vials the lab will analyze.
  - If the pH paper reads above 2, then add another gram of acid and repeat this procedure one more time.

Now that the soil chemistry has been determined, the actual sampling can occur. The procedure stated below assumes the correct vials are used based on the guidance discussed.

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### Sample Preservation Steps

1. Wear gloves during all handling of pre-weighed vials.
2. Quickly collect a 5g sample using a cut off plastic syringe or other coring device designed to deliver 5g of soil from a freshly exposed surface of soil.
3. Carefully wipe exterior of sample collection device with a clean paper towel.
4. Quickly transfer to the appropriate VOA vial, extruding with caution so that the solution does not splash out of the vial.
5. Add more acid if necessary (this is based on the buffering testing discussed in the previous section).
6. Use the paper toweling and quickly remove any soil off the vial threads.
7. Cap vial and weigh the jar to the nearest 0.01g.
8. Record exact weight on sample label.
9. Repeat sampling procedure for the duplicate VOA vial.
10. Weigh the vial containing methanol preservative to the nearest 0.01g. If the weight of the vial with methanol varies by more than 0.01g from the original weight recorded on the vial, discard the vial. If the weight is within tolerance, it can be used for soil preservation below.
11. Take the empty jar or the jar that contains the methanol preservative.
12. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size is dependent on who is doing the sampling and who is doing the laboratory analysis.
13. Carefully wipe the exterior of the collection device with a clean paper towel.
14. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol, be careful not to splash the methanol outside of the vial. Again, the type of jar received is dependent on who is doing the laboratory analysis.
15. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided, using only one vial of methanol preservative per sample jar.

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16. Use the paper toweling and remove any soil off of the vial threads and cap the jar.
17. Weigh the jar with the soil in it to the nearest 0.01g and record the weight on the sample label.
18. Collect dry weight sample-fill container.
19. Store samples at 4° Celsius.
20. Ship sample containers with plenty of ice in accordance with Department of Transportation (DOT) regulations (CORROSIVE, FLAMMABLE LIQUID, POISON) to the laboratory.

### 5.2.2.3 EN CORE™ SAMPLER COLLECTION FOR HIGH LEVEL ANALYSES (≥200 UG/KG)

#### EN CORE™ Sampling Equipment Requirements

The following equipment is required for high-level analysis:

- One 25g sampler or one 5g sampler (The sampler size used will be dependent on who is doing the sampling and who is doing the laboratory analysis).
- One dry weight cup.
- One T-handle.
- Paper towels.

#### EN CORE™ Sampling Steps for High Level Analysis

1. Remove sample and cap from package and attach T-handle to sampler body.
2. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.
3. Use paper toweling to quickly wipe the sampler head so that the cap can be tightly attached.
4. Push cap on with a twisting motion to attach cap.
5. Fill out a sample label and attach to sampler.
6. Collect dry weight sample.
7. Store samplers at 4° Celsius.
8. Ship sample containers with plenty of ice to the laboratory within 40 hours of collection.

**NOTE:** Verify state requirements for extraction/holding times.

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### 5.2.2.4 METHANOL PRESERVATION SAMPLING FOR HIGH LEVEL ANALYSES ( $\geq 200$ UG/KG)

#### Methanol Preservation Sampling Equipment Requirements

- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a pre-weighed vial that contains methanol (laboratory grade).
- One dry weight cup.
- Weighing balance that accurately weighs to 0.01g (with accuracy of  $\pm 0.1$ g).
- Set of balance weights used in daily balance calibration.
- Latex gloves.
- Paper towel.
- Cutoff plastic syringe or other coring device to deliver 5g or 25g of soil.

#### Sampling Preservation Steps

1. Wear gloves during all handling of pre-weighed vials.
2. Weigh the vial containing methanol preservative to the nearest 0.01g. If the weight of the vial with methanol varies by more than 0.01g from the original weight recorded on the vial, discard the vial. If the weight is within tolerance, it can be used for soil preservation/collection below.
3. Take the empty jar or the jar that contains the methanol preservative.
4. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size used is dependent on who is doing the sampling and who is doing the laboratory analysis.
5. Carefully wipe the exterior of the collection device with a clean paper towel.
6. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol, be careful not to splash the methanol outside of the vial. Again, the type of jar used is dependent on who is doing the laboratory analysis.
7. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided, using only one vial of methanol preservative per sample jar.
8. Using the paper toweling, remove any soil off of the vial threads and cap the jar.
9. Weigh the jar with the soil in it to the nearest 0.01g and record the weight on the sample label.
10. Collect dry weight sample-fill container.



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11. Store samples at 4° Celsius.

12. Ship sample containers with plenty of ice in accordance with DOT regulations (CORROSIVE, FLAMMABLE LIQUID, POISON) to the laboratory.

### **5.2.3 Method for Collecting Samples for Nonvolatile Organic or Inorganic Compound Analysis**

The requirements for collecting samples of surface soil for nonvolatile organic or inorganic analyses are as follows:

1. Label each sample container with the appropriate information. Secure the label by covering it with a piece of clear tape.
2. Use a decontaminated stainless steel or Teflon®-lined trowel or spoon to obtain sufficient sample from the required interval and subsampling points, if necessary, to fill the specified sample containers.
3. Empty the contents of each fill of the sampling device directly into a clean stainless steel or Teflon®-lined tray or bowl.
4. Homogenize the sample by mixing with a spoon, spatula, or trowel.
5. Use the spoon, spatula, or trowel to distribute the uniform mixture into the labeled sample containers. Fill organic sample containers first, then inorganics.
6. Secure the appropriate cap on each container immediately after filling it.
7. Wipe the sample containers with a clean Kimwipe or paper towel.
8. Place sample containers in individual zip-top plastic bags and seal the bags.
9. Pack all samples as required. Include properly completed documentation, and affix custody seals to the cooler lid.
10. Decontaminate sampling equipment according to CDM Federal SOP 4-5.

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### 5.2.4 Manual (Hand) Augering Using a Tube Sampler With Liner

The following steps must be followed when collecting hand-augered samples using a tube sampler with liner:

1. Auger to the depth required for sampling. Place cuttings on the plastic sheeting as specified in the site-specific plans. If possible, lay out the cuttings in stratigraphic order.
2. Throughout augering, make detailed notes concerning the geologic features of the soil or sediments in the field logbook.
3. Cease augering when the top of the specified sampling depth has been reached. Remove the auger from the hole and decontaminate.
4. Prepare a decontaminated tube sampler by installing a decontaminated liner in the auger tube.
5. Obtain the sample and retrieve the auger. Remove the liner from the tube and immediately cover ends with Teflon® tape and cap the ends of the tube. Seal the caps with waterproof tape.
6. Label the sealed liners as required in the site-specific plans. Mark the top and bottom of the sample on the outside of the liner. Indicate boring/well number and depth on outside of liner.
7. Wipe sealed liners with a clean Kimwipe or paper towel.
8. Place sealed liners in zip-top plastic bags and seal the bags. Pack samples in a chilled cooler.
9. Proceed with further sampling, as required by the site-specific plans.
10. When sampling is complete, dispose of cuttings, plastic sheeting, etc., as specified in the site-specific plans.
11. Decontaminate all equipment according to CDM Federal SOP 4-5.
12. Complete the field logbook entry and other forms, being sure to record all relevant information before leaving the site.
13. Properly package all samples for shipment and complete all necessary sample shipment documentation. Remand custody of samples to the appropriate personnel. See CDM Federal SOPs 1-2 and 2-1 or site-specific plans.

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### 5.2.5 Split-Spoon Sampling

The following steps must be followed when collecting split-spoon samples:

1. Remove any pavement and subbase material from an area of twice the bit diameter, if necessary.
2. The drilling rig will be decontaminated at a separate location prior to drilling, per CDM Federal SOP 4-5 or the site-specific decontamination procedures.
3. Attach the hollow-stem auger with the cutting head, plug, and center rod(s) to the drill rig.
4. Begin drilling and proceed to the first designated sample depth, adding auger flight(s) as necessary.
5. Slightly raise the auger flight(s) to disengage the cutting head, and rotate the auger without advancement to clean cuttings from the bottom of the hole.
6. Remove the plug and center rods.
7. Install a decontaminated split spoon on the center rod(s) and insert it into the hollow-stem auger. Connect the hammer assembly and lightly tap the rods to seat the drive shoe at the top of undisturbed soil or sediment.
8. Mark the center rod in 6-inch increments from the top of the auger flight(s).
9. Drive the spoon using the hammer. Use a full 30-inch drop as specified by the American Society for Testing and Materials (ASTM) Method D-1586. Record the number of blows required to drive the spoon or tube through each 6-inch increment.
10. Cease driving when the full length of the spoon has been driven or upon refusal. Refusal occurs when little (<1 inch) or no progress is made for 50 blows of the hammer.
11. Pull the spoon or tube free by using upswings of the hammer to loosen the sampler. Pull out the center rod and spoon or tube.
12. Unscrew the split-spoon assembly from the center rod and place it on the plastic sheeting.
13. Remove the drive shoe and head assembly. If necessary, tap the split-spoon assembly with a hammer to loosen threaded couplings.
14. With the drive shoe and head assembly off, open (split) the spoon, being careful not to disturb the sample.

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15. Label sample containers with appropriate information. Secure the label, covering it with a piece of clear tape.
16. If VOC analyses are to be conducted on the soil sample, place that sample in its sample container immediately after opening the spoon, filling the sample bottle completely. Seal the container immediately, then describe it in the field logbook and/or associated forms. Record the sample identification number, depth from which the sample was taken, and the analyses to be performed on the samples in the field logbook and on the appropriate forms.
17. Remaining sample should be homogenized prior to placing samples in appropriate containers. Label containers as required.
18. Wipe containers with a clean Kimwipe or paper towel.
19. Place containers in zip-top plastic bags and seal the bags. Pack samples in a chilled cooler.
20. Continue to advance the borehole to the next sampling point. Collect samples as outlined above.
21. When sampling is complete, remove the drilling rig to the heavy equipment decontamination area.
22. Dispose of cuttings, plastic sheeting, etc., as specified in the site-specific plans. Backfill bore hole as specified in project-specific plans.
23. Decontaminate split spoons and other small sampling equipment according to CDM Federal SOP 4-5 before proceeding to other sampling locations.
24. Complete the field logbook entry and other forms, being sure to record all relevant information before leaving the site.
25. Properly package all samples for shipment to laboratories and complete all necessary sample shipment documentation. Remand custody of the samples to the appropriate personnel. See CDM Federal SOPs 1-2 and 2-1 or site-specific plans.

### 5.2.6 Split-Spoon Sampling Using Liners

The following steps must be followed when collecting samples with lined split spoons:

1. Remove any pavement and sub base material from an area of twice the bit diameter, if necessary.
2. The drilling rig will be decontaminated at a separate location prior to drilling.
3. Attach the hollow-stem auger with the cutting head and center rod(s).

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4. Begin drilling and proceed to the first designated sample depth, adding auger flight(s) as necessary.
5. Slightly raise the auger flight(s) to disengage the cutting head, and rotate the auger without advancement to clean cuttings from the bottom of the hole.
6. Remove the plug and center rods.
7. Install decontaminated liners in the split-spoon barrel.
8. Install a decontaminated split spoon on the center rod(s) and insert it into the hollow-stem auger. Connect the hammer assembly and lightly tap the rods to seat the drive shoe at the top of undisturbed soil or sediment.
9. Mark the center rod in 6-inch increments from the top of the auger flight(s).
10. Drive the spoon using the hammer. Use a full 30-inch drop as specified by ASTM Method D-1586. Record the number of blows required to drive the spoon or tube through each 6-inch increment.
11. Cease driving when the full length of the spoon has been driven or upon refusal. Refusal occurs when little (<1 inch) or no progress is made after 50 blows of the hammer.
12. Pull the spoon or tube free by using upswings of the hammer to loosen the sampler. Pull out the center rod and spoon or tube.
13. Unscrew the split-spoon assembly from the center rod and place it on the plastic sheeting.
14. Remove the drive shoe and head assembly. If necessary, tap the split-spoon assembly with a hammer to loosen threaded couplings.
15. With the drive shoe and head assembly off, open (split) the spoon and remove the liners without disturbing the sample.
16. Immediately install Teflon® tape over the ends of the liners, cap the liners, and seal the caps over the ends of the liner with waterproof tape. Label the samples as required by the site-specific plans. Mark the top and bottom of each sample on the outside of each liner. Indicate boring/well number and depth on outside of liner.
17. Wipe sealed liners with a clean Kimwipe or paper towel.
18. Place sealed liners in zip-top plastic bags and seal the bags. Pack samples in a chilled cooler.

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19. In the field logbook and on the boring log, describe sample lithology by observing cuttings and the bottom end of the sample in the liner.
20. Continue to advance the borehole to the next sampling point. Collect samples as outlined above.
21. When sampling is complete, remove the drilling rig to the heavy equipment decontamination site.
22. Dispose of cuttings, plastic sheeting, etc., as specified in the site-specific plans.
23. Decontaminate split spoons and other small sampling equipment before proceeding to other sampling locations as required by the CDM Federal SOP 4-5.
24. Complete the field logbook entry, and other forms, being sure to record all relevant information before leaving the site.
25. Properly package all samples for shipment to laboratories and complete all necessary sample shipment documentation. Remand custody of the samples to the appropriate personnel. See CDM Federal SOPs 1-2 and 2-1 or site-specific plans.

### **5.2.7 Shelby Tube Sampling**

The following steps must be followed when collecting samples using the Shelby tube:

1. Remove any pavement and sub base material from an area of twice the bit diameter, if necessary.
2. The drilling rig will be decontaminated at a separate location prior to drilling.
3. Attach the hollow-stem auger with the cutting head, plug, and center rod(s).
4. Begin drilling and proceed to the first designated sample depth, adding auger flight(s) as necessary.
5. Slightly raise the auger flight(s) to disengage the cutting head, and rotate the auger without advancement to clean cuttings from the bottom of the hole.
6. Remove the plug and center rods.
7. Attach a head assembly to a decontaminated Shelby tube. Attach the Shelby tube assembly to the center rods.
8. Lower the Shelby tube and center rods into the hollow-stem augers and seat it at the bottom. Be sure to leave 30 inches or more of center rod above the lowest point to the hydraulic piston's extension.

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9. Use the rig's hydraulic drive to push the Shelby tube into undisturbed soil. The tube should be pushed with a steady force. Note the pressure used to push the Shelby tube in the field logbook.
10. When the Shelby tube has been advanced its full length or to refusal, back off the hydraulic pistons. Attach a hoisting plug to the upper end of the center rod, twist to break off the sample, and pull the apparatus out of the hole with the rig winch.
11. Retrieve the Shelby tube to the surface, detach it from the center rod, and remove the head assembly.
12. Since the typical intent of Shelby tube sampling is for engineering purposes and an undisturbed sample is required, the tube ends should be sealed immediately. Sealing is accomplished by filling any void space in the tube with beeswax, then placing caps on the ends of the tube and taping caps into place. The top and bottom ends of the tube should be marked and the tube transported to the laboratory in an upright position. Indicate boring/well number and depth on outside of liner.
13. Wipe sealed tubes with a clean Kimwipe or paper towel.
14. Place sealed tubes in zip-top plastic bags and seal bags. Pack samples in a chilled cooler.
15. Continue to advance the borehole to the next sampling point. Collect samples as outlined above.
16. When sampling is complete, remove the drilling rig to the heavy equipment decontamination area.
17. Dispose of cuttings, plastic sheeting, etc., as specified in the site-specific plans.
18. Complete the field logbook entry, being sure to record all relevant information before leaving the site. These methods may be used if directed by the EPA region, client or governing sample plan.

### 6.0 RESTRICTIONS/LIMITATIONS

Basket or spring retainers may be needed for split-spoon sampling in loose, sandy soils.

Shelby tubes may not retain the sample in loose, sandy soils.

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### 7.0 REFERENCES

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# PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLES

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**1.0 PACKAGING AND SHIPPING OF ALL SAMPLES** – This standard operating procedure (SOP) applies to the packaging and shipping of all environmental samples. If the sample is preserved or radioactive, the following sections may also be applicable.

Section 2.0 – Packaging and Shipping of Samples Preserved with Hexane

Section 3.0 – Packaging and Shipping of Samples Preserved with Sodium Hydroxide

Section 4.0 – Packaging and Shipping of Samples Preserved with Hydrochloric Acid

Section 5.0 – Packaging and Shipping of Samples Preserved with Nitric Acid

Section 6.0 – Packaging and Shipping of Samples Preserved with Sulfuric Acid

Section 7.0 – Packaging and Shipping of Limited Quantity Radioactive Samples

## 1.1 OBJECTIVE

The objective of this SOP is to outline the requirements for the packaging and shipment of environmental samples.

## 1.2 BACKGROUND

### 1.2.1 Definitions

**Environmental Sample** – An environmental sample is any sample that has less than reportable quantities for any hazardous constituents according to Department of Transportation (DOT) regulations promulgated in 49 CFR - Part 172.

**Custody Seal** – A custody seal is a narrow adhesive-backed seal that is applied to individual sample containers and/or the sample shipping container (i.e. cooler) before offsite shipment. Custody seals are used as a protective mechanism to ensure that sample integrity is not compromised during transportation from the field to the analytical laboratory.

**Secondary Containment** – A secondary containment is the container that the sample is shipped in (i.e., plastic overpackaging if liquid sample is collected in glass).

**Exempted Quantity** – Exempted quantity is the amount of hazardous material that does not fall under DOT/IATA/ICAO regulations. This exemption is very difficult to meet; most shipments will be made under limited quantity.

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Limited Quantity – Limited quantity is the maximum amount of a hazardous material for which there is a specific labeling or packaging exception.

Performance Testing – Performance testing is the required testing of outer packaging. These tests include the drop and stacking test.

Qualified Shipper – A qualified shipper is a person who has been adequately trained to perform the functions of shipping hazardous materials.

### **1.2.2 Discussion**

Proper packaging and shipping is necessary to ensure the protection of the integrity of environmental samples shipped for analysis.

### **1.2.3 Associated Procedure**

- CDM Federal SOP 1-2, Sample Custody

## **1.3 RESPONSIBILITIES**

**Field Team Leader (FTL)** - The field team leader is responsible for ensuring that packaging and sampling procedures are conducted in accordance with this SOP. The field team leader is also responsible for ensuring that CDM Federal properly coordinates laboratory analysis of samples.

## **1.4 REQUIRED EQUIPMENT**

- Coolers with return address of CDM Federal office
- Heavy-duty plastic garbage bags
- Plastic Ziploc®-type bags, small and large
- Clear tape
- Fiber tape – nylon reinforced strapping tape
- Duct tape
- Vermiculite (or equivalent)\*
- Bubble wrap (optional)
- Ice
- Custody seals
- Completed chain-of-custody record or CLP custody records, if applicable
- Completed bill of lading
- "This End Up" and directional arrow labels

\* Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

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### **1.5 PROCEDURES**

The following steps must be followed when packing sample bottles and jars for shipment:

1. Verify the samples undergoing shipment meet the definition of "Environmental Sample" and are not a hazardous material as defined by DOT. Professional judgment and/or consultation with the appropriate health and safety coordinator or the health and safety manager should be observed.
2. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape. Line the cooler with a large heavy-duty plastic garbage bag.
3. Be sure the caps on all bottles are tight (will not leak); check to see that labels and chain-of-custody records are completed properly (SOP 1-2, Sample Custody).
4. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Up to three VOA vials may be packed in one bag. Bottles may be wrapped in bubble wrap. Optionally, place three to six VOA vials in a quart metal can and then fill the can with vermiculite or equivalent. Note: Trip blanks must be included in coolers containing VOA samples.
5. Place 2 to 4 inches of vermiculite (or equivalent) into a cooler that has been lined with a garbage bag, and then place the bottles and cans in the bag with sufficient space to allow for the addition of more packing material between the bottles and cans. It is preferable to place glass sample bottles and jars into the cooler vertically. Due to the strength properties of a glass container, there is much less chance for breakage when the container is packed vertically rather than horizontally.
6. Put ice in large plastic zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place the ice bags on top of and/or between the samples. Several bags of ice are required (dependant on outdoor temperature, staging time, etc.) to maintain the cooler temperature at approximately 4° centigrade. Fill all remaining space between the bottles or cans with packing material. Securely fasten the top of the large garbage bag with fiber or duct tape.
7. Place the completed chain-of-custody record or the CLP traffic report form (if applicable) for the laboratory into a plastic zip-top bag, seal the bag, tape the bag to the inner side of the cooler lid and close the cooler.
8. The cooler lid shall be secured with nylon reinforced strapping tape by wrapping each end of the cooler a minimum of two times. Attach a completed chain-of-custody seal across the hinges of the cooler on opposite sides. The custody seals should be affixed to the cooler with half of the seal on the strapping tape so that the cooler cannot be opened without breaking the seal. Complete two more wraps around with fiber tape and place clear tape over the custody seals.

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9. The shipping container lid must be marked "THIS END UP" and arrow labels that indicate the proper upward position of the container should be affixed to the cooler. A label containing the name and address of the shipper (CDM Federal) shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Air Craft, Flammable Solids, etc.) are not permitted on the outside of containers used to transport environmental samples and shall not be used. The name and address of the laboratory shall be placed on the container, or when shipping by common courier, the bill of lading shall be completed and attached to the lid of the shipping container.

### **1.6 RESTRICTIONS/LIMITATIONS**

The holding times for the samples packed for shipment must not be exceeded. It is recommended that samples be packed in time to be shipped nightly for overnight delivery. Use caution when shipping samples for weekend delivery; make arrangements with the laboratory before sending samples.

## **2.0 PACKAGING AND SHIPPING OF SAMPLES PRESERVED WITH HEXANE**

### **2.1 OBJECTIVE**

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

### **2.2 BACKGROUND**

#### **2.2.1 Definitions**

Section 1.2.1 defines the terms relevant to this section.

#### **2.2.2 Transportation**

This section was prepared for the shipment of hexane-preserved samples.

#### **2.2.3 Containers**

- 40 ml glass VOA vials (up to 1L per outer package)

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### **2.3 RESPONSIBILITY**

It is the responsibility of the qualified shipper to ensure that each shipment contains no more than the maximum of 24 VOA vials for a total liquid volume of 1 liter and that the shipment is packaged according to LATA/ICAO packaging instruction Y305 for limited quantities of hexane.

### **REQUIRED EQUIPMENT**

- Outer packaging (for limited quantities) insulated cooler that has passed the performance test
- Garbage bags
- Clear tape
- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (or equivalent)\*
- Bubble wrap
- Ice
- Chain-of-custody seals
- Chain-of-custody form
- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 3 flammable liquid labels
- Orientation labels
- Consignor/consignee labels

\* Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

### **2.5 PACKAGING**

The following steps are to be followed when packaging limited quantity samples shipments.

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials

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- Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody)
- Wrap each container (40 ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble wrapped container into a 2.7 mil Ziploc®-type bag, removing trapped air.
- Place wrapped containers inside a polyethylene bottle filled with vermiculite; seal the bottle. (Maximum of 4 VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

### **HEXANES MIXTURE**

**UN1208**

**LTD. QTY.**

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Flammable Liquid label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marketing locations is shown in Figure 1.

**NOTE:** No marking or labeling can be obscured by strapping or duct tape.

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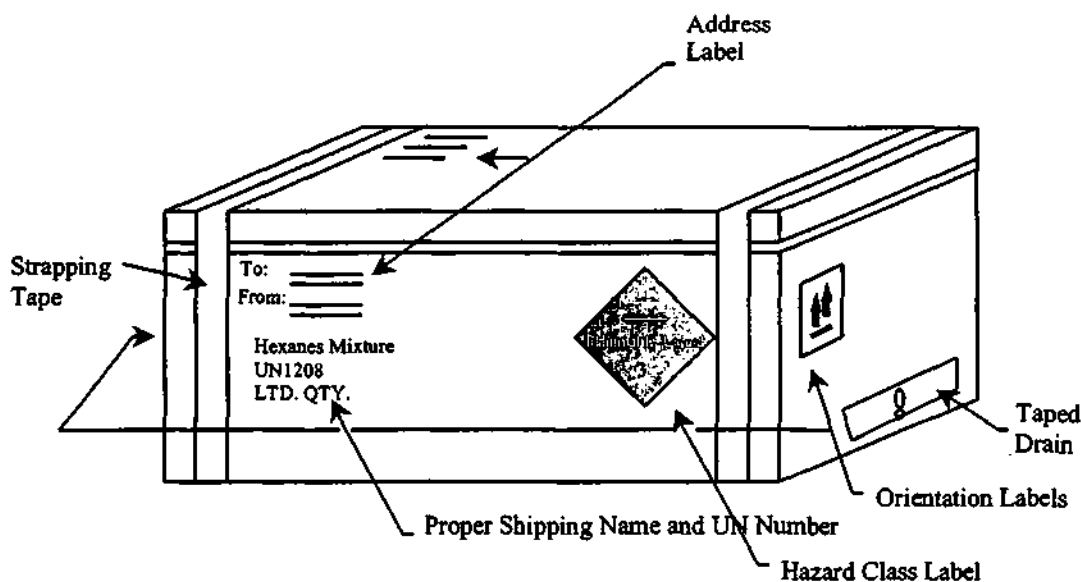
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**NOTE:** The inner packaging of dangerous goods may be placed into the designated cooler for shipment. Other non-regulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

**Figure 1 Example of Cooler Label/Marking Locations**



### 3.0 PACKAGING AND SHIPPING OF SAMPLES PRESERVED WITH SODIUM HYDROXIDE

#### 3.1 OBJECTIVE

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

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### 3.2 BACKGROUND

#### 3.2.1 Definitions

Section 1.2.1 defines the terms relevant to this section.

#### 3.2.2 Transportation

This section was prepared for the shipment of sodium hydroxide (NaOH) preserved samples.

#### 3.2.3 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Exempted Quantities of Preservatives

Preservative		Desired in Final Sample		Quantity of Preservative (ml) for Specified Container				
				40 ml	125 ml	250 ml	500 ml	1 L
NaOH	30%	pH >12	Conc. 0.08%		.25	0.5	1	2

5 drops = 1 ml

### 3.3 RESPONSIBILITY

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

### REQUIRED EQUIPMENT

- Outer packaging (for limited quantities) insulated cooler that has passed the performance test.
- Garbage bags
- Clear tape
- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (or equivalent)\*
- Bubble wrap (optional)
- Ice
- Custody seals
- Chain-of-custody form



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- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

\* Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

### **3.5 PACKAGING**

Samples containing NaOH as a preservative that exceed the exempted concentration of 0.08 percent (2 ml of a 30 percent per liter) will be shipped as a limited quantity per packing instruction Y809 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited quantity samples shipments.

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials
  - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody)
- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble wrapped container into a 2.7 mil Ziploc®-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.

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- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

**SODIUM HYDROXIDE SOLUTION  
UN1824  
LTD. QTY.**

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marketing locations is shown in Figure 1.

**NOTE:** Samples meeting the exemption concentration of 0.08 percent NaOH by weight will be shipped as non-regulated or non-hazardous.

**NOTE:** No marking or labeling can be obscured by strapping or duct tape.

**NOTE:** The inner packaging of dangerous goods may be placed into the designated cooler for shipment. Other non-regulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

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### 4.0 PACKAGING AND SHIPPING OF SAMPLES PRESERVED WITH HYDROCHLORIC ACID

#### 4.1 OBJECTIVE

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

#### 4.2 BACKGROUND

##### 4.2.1 Definitions

Section 1.2.1 defines the terms relevant to this section.

##### 4.2.2 Transportation

This section was prepared for the shipment of hydrochloric acid (HCl) preserved samples.

##### 4.2.3 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Exempted quantities of preservatives

Preservative		Desired in Final Sample		Quantity of Preservative (ml) for Specified Container				
				40 ml	125 ml	250 ml	500 ml	1 L
HCl	2N	pH <2	Conc. 0.04%	.2	.5	1		

5 drops = 1 ml

#### 4.3 RESPONSIBILITY

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

#### 4.4 REQUIRED EQUIPMENT

- Outer packaging (for limited quantities) insulated cooler that has passed the performance test.
- Garbage bags
- Clear tape

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- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (or equivalent)\*
- Bubble wrap
- Ice
- Custody seals
- Chain-of-custody form
- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

\* Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

### 4.5 PACKAGING

The following steps are to be followed when packaging limited quantity samples shipments.

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials
  - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody)
- Wrap each container (40 ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble wrapped container into a 2.7 mil Ziploc®-type bag, removing trapped air.
- Place wrapped containers inside a polyethylene bottle filled with vermiculite; seal the bottle. (Maximum of 4 VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.

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- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

### **HYDROCHLORIC ACID SOLUTION**

**UN1789**

**LTD. QTY.**

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marketing locations is shown in Figure 1.

**NOTE:** Samples meeting the exemption concentration of 0.04 percent HCl by weight will be shipped as non-regulated or non-hazardous.

**NOTE:** No marking or labeling can be obscured by strapping or duct tape.

**NOTE:** The inner packaging of dangerous goods may be placed into the designated cooler for shipment. Other non-regulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

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- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

### 5.0 PACKAGING AND SHIPPING OF SAMPLES PRESERVED WITH NITRIC ACID

#### 5.1 OBJECTIVE

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

#### 5.2 BACKGROUND

##### 5.2.1 Definitions

Section 1.2.1 defines the terms relevant to this section.

##### 5.2.2 Transportation

This section was prepared for the shipment of nitric acid ( $\text{HNO}_3$ ) preserved samples.

##### 5.2.3 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Exempted quantities of preservatives

Preservative		Desired in Final Sample		Quantity of Preservative (ml) for Specified Container				
		pH	Conc.	40 ml	125 ml	250 ml	500 ml	1 L
$\text{HNO}_3$	6N	<2	0.15%		2	4	5	8

5 drops = 1 ml

#### 5.3 RESPONSIBILITY

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

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### **5.4 REQUIRED EQUIPMENT**

- Outer packaging (for limited quantities) insulated cooler that has passed the performance test.
- Garbage bags
- Clear tape
- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (or equivalent)\*
- Bubble wrap (optional)
- Ice
- Custody seals
- Chain-of-custody form
- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

\* Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

### **5.5 PACKAGING**

Samples containing  $\text{HNO}_3$  as a preservative that exceed the exempted concentration of 0.15%  $\text{HNO}_3$  will be shipped as a limited quantity per packing instruction Y807 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited quantity samples shipments.

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials
  - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody)

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- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble wrapped container into a 2.7 mil Ziploc®-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

### **NITRIC ACID SOLUTION (with less than 20%)**

**UN2031**

**LTD. QTY.**

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marketing locations is shown in Figure 1.

**NOTE:** Samples meeting the exemption concentration of 0.15 percent  $\text{HNO}_3$  by weight will be shipped as non-regulated or non-hazardous.

**NOTE:** No marking or labeling can be obscured by strapping or duct tape.



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**NOTE:** The inner packaging of dangerous goods may be placed into the designated cooler for shipment. Other non-regulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

### 6.0 PACKAGING AND SHIPPING OF SAMPLES PRESERVED WITH SULFURIC ACID

#### 6.1 OBJECTIVE

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

#### 6.2 BACKGROUND

##### 6.2.1 Definitions

Section 1.2.1 defines the terms relevant to this section.

##### 6.2.2 Transportation

This section was prepared for the shipment of sulfuric acid ( $H_2SO_4$ ) preserved samples.

##### 6.2.3 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Exempted quantities of preservatives

Preservative		Desired in Final Sample		Quantity of Preservative (ml) for Specified Container				
				pH	Conc.	40 ml	125 ml	250 ml
H <sub>2</sub> SO <sub>4</sub>	37N	<2	0.35%	.1	.25	0.5	1	2

5 drops = 1 ml

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### **6.3 RESPONSIBILITY**

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

### **6.4 REQUIRED EQUIPMENT**

- Outer packaging (for limited quantities) insulated cooler that has passed the performance test.
- Garbage bags
- Clear tape
- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (or equivalent)\*
- Bubble wrap
- Ice
- Custody seals
- Chain-of-custody form
- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

\* Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

### **6.5 PACKAGING**

Samples containing  $H_2SO_4$  as a preservative that exceed the exempted concentration of 0.35 percent will be shipped as a limited quantity per packing instruction Y809 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited quantity samples shipments.

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection

## **PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLES**

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- Sample location
- Sample identification number
- Collector's initials
- Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody)
- Wrap each glass container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble wrapped container into a 2.7 mil Ziploc®-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

### **SULFURIC ACID SOLUTION**

**UN2796**

**LTD. QTY.**

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marketing locations is shown in Figure 1.

## **PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLES**

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**NOTE:** Samples meeting the exemption concentration of 0.35 percent  $H_2SO_4$  by weight will be shipped as non-regulated or non-hazardous.

**NOTE:** No marking or labeling can be obscured by strapping or duct tape.

**NOTE:** The inner packaging of dangerous goods may be placed into the designated cooler for shipment. Other non-regulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

### **7.0 PACKAGING AND SHIPPING OF LIMITED QUANTITY RADIOACTIVE SAMPLES**

#### **7.1 OBJECTIVE**

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

#### **7.2 BACKGROUND**

##### **7.2.1 Definitions**

Section 1.2.1 defines the terms relevant to this section.

##### **7.2.2 Transportation**

This section was prepared for the shipment of environmental samples containing radioactive materials in limited quantities.

##### **7.2.3 Containers**

The inner packaging containers that may be used for these shipments include:

- Any size sample container

## PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLES

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### 7.3 DESCRIPTION/RESPONSIBILITIES

- The qualified shipper will ship all samples that meet the Class 7 definition of radioactive materials and meet the activity requirements specified in Table 7 of 49 CFR 173.425, as Radioactive Materials in Limited Quantity. The qualified shipper will verify that all packages and their contents meet the requirements of 49 CFR 173.421, "Limited Quantities of Radioactive Materials."
- The packaging used for shipping will meet the general requirements for packaging and packages specified in 49 CFR 173.24 and the general design requirements provided in 173.410. These standards state that a package must be capable of withstanding the effects of any acceleration, vibration, or vibration resonance that may arise under normal condition of transport without any deterioration in the effectiveness of the closing devices on the various receptacles or in the integrity of the package as a whole and without loosening or unintentionally releasing the nuts, bolts, or other securing devices even after repeated use.
- If the shipment is from a Department of Energy (DOE) facility, radiological screenings will be completed on all samples taken. The qualified shipper will review the results of each screening (alpha, beta, and gamma speciation). Samples will not be shipped offsite until the radiological screening has been performed.
- The total activity for each package will not exceed the relevant limits listed in Table 7 of 49 CFR 173.425. The  $A_2$  value of the material will be calculated based on all radionuclides found during previous investigations (if any) in the area from which the samples are derived. The  $A_2$  values to be used will be the most restrictive of all potential radionuclides as listed in 49 CFR 173.435.
- The radiation level at any point on the external surface of the package bearing the sample(s) will not exceed 0.005 mSv/hour (0.5 mrem/hour). These will be verified by dose and activity monitoring prior to shipment of the package.
- The removable radioactive surface contamination on the external surface of the package will not exceed the limits specified in 49 CFR 173.443(a). CDM Federal will use the DOE-established free release criteria for removable surface contamination of less than 20 dpm/100 cm<sup>2</sup> (alpha) and 1000 dpm/100 cm<sup>2</sup> (beta/gamma). It should be noted that these values are *more conservative than the DOT requirements for removable surface contamination*.
- The qualified shipper will verify that the outside of the inner packaging is marked "Radioactive".
- The qualified shipper will verify that the excepted packages prepared for shipment under the provisions of 49 CFR 173.421 have a notice enclosed, or shown on the outside of the package, that reads, "This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910".

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### **7.4 REQUIRED EQUIPMENT**

- Cooler or other acceptable outer packaging
- Garbage bags
- Clear tape
- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (for water samples) or equivalent\*
- Bubble wrap (optional)
- Ice (if necessary)
- Custody seals
- Chain-of-custody form
- Survey documentation/radiation screening results (if shipping from DOE or radiological sites)
- Orientation labels
- Exempted quantities label
- Consignor/consignee labels

\* Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

### **7.5 PACKAGING**

The following steps are to be followed when packaging limited quantity samples shipments.

- The cooler is to be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
  - Project name
  - Project number
  - Date and time of sample collection
  - Sample location
  - Sample identification number
  - Collector's initials
- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.

## PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLES

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- Place sufficient amount of vermiculite, or approved packaging material, in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- If required, place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- Place a label marked "Radioactive" on the outside of the sealed bag.
- Enclose a notice that includes the name of the consignor or consignee and the following statement: "This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910.
- The maximum weight of the package shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- If a cooler is used, wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix package orientation labels on two opposite sides of the cooler/package.
- Affix a completed Excepted Quantities label to the side of the cooler/package.
- Secure any marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of the cooler labeling/marketing is shown in Figure 2.

**NOTE:** No marking or labeling can be obscured by strapping or duct tape.

- Complete the Shipment Quality Assurance Checklist (Appendix B).

**NOTE:** Except as provided in 49 CFR 173.426, the package will not contain more than 15 grams of  $^{235}\text{U}$ .

**NOTE:** A declaration of dangerous goods is not required.

## PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLES

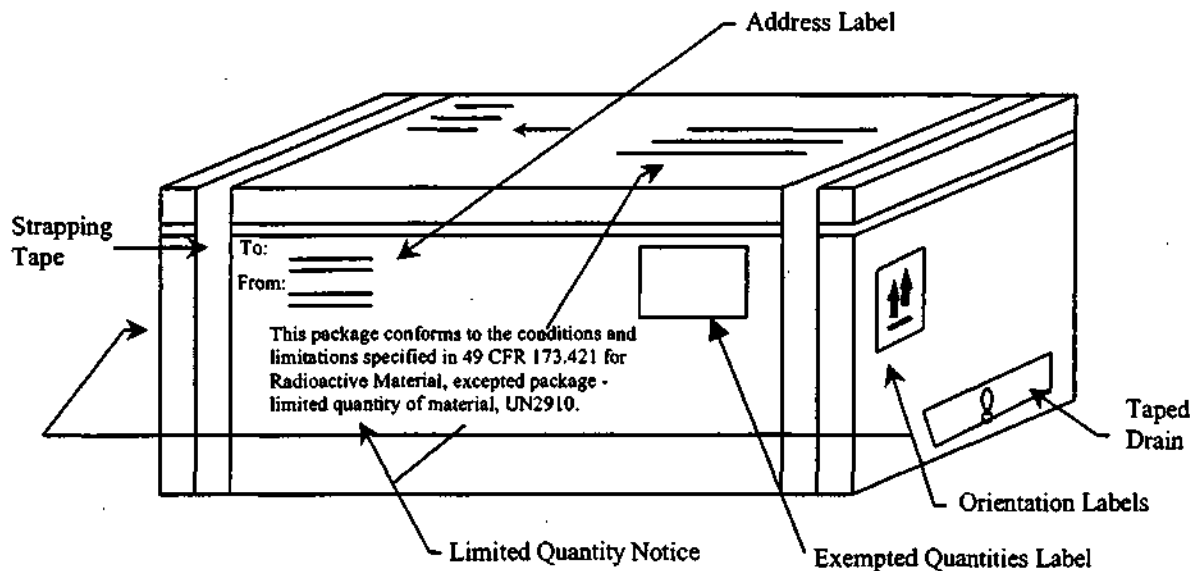
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**Figure 2 Radioactive Material - Limited Quantity Cooler Marking Example**



### 8.0 REFERENCES

U.S. Environmental Protection Agency, *Sampler's Guide to the Contract Laboratory Program*, EPA/540/P-90/006, December 1990.

U.S. Environmental Protection Agency, Region IV, *Standard Operating Procedures and Quality Assurance Manual*, February 1991.



# PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLES

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## APPENDIX A

### Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity

#### Sample Packaging

Yes No N/A

☐ ☐ ☐

The VOA vials are wrapped in bubble wrap and placed inside a Ziploc®-type bag.

☐ ☐ ☐

The VOA vials are placed into a polyethylene bottle, filled with vermiculite, and tightly sealed.

☐ ☐ ☐

The drain plug is taped inside and outside to ensure control of interior contents.

☐ ☐ ☐

The samples have been placed inside garbage bags with sufficient bags of ice to preserve samples at 4°C.

☐ ☐ ☐

The cooler exceeds the 66-pound limit for limited quantity shipment.

☐ ☐ ☐

The garbage bag has been sealed with tape (or tied) to prevent movement during shipment.

☐ ☐ ☐

The chain-of-custody has been secured to the interior of the cooler lid.

☐ ☐ ☐

The cooler lid and sides have been taped to ensure a seal.

☐ ☐ ☐

The custody seals have been placed on both the front and back hinges of the cooler, using waterproof tape.

#### Air Waybill Completion

Yes No N/A

☐ ☐ ☐

Section 1 has the shipper's name, company and address; the account number, date, internal billing reference number; and the telephone number where the shipper can be reached.

☐ ☐ ☐

Section 2 has the recipient's name and company along with a telephone number where they can be reached.

☐ ☐ ☐

Section 3 has the **Bill Sender** box checked.

☐ ☐ ☐

Section 4 has the **Standard Overnight** box checked.

☐ ☐ ☐

Section 5 has the **Deliver Weekday** box checked.

☐ ☐ ☐

Section 6 has the number of packages and their weights filled out. Was the total of all packages and their weights figured up and added at the bottom of Section 6?

☐ ☐ ☐

Under the **Transport Details** box, the **Cargo Aircraft Only** box is obliterated, leaving only the **Passenger and Cargo Aircraft** box.

☐ ☐ ☐

Under the **Shipment Type**, the **Radioactive** box is obliterated, leaving only the **Non-Radioactive** box.

☐ ☐ ☐

Under the **Nature and Quantity of Dangerous Goods** box, the **Proper Shipping Name, Class or Division, UN or ID No., Packing Group, Subsidiary Risk, Quantity and Type of Packing, Packing Instructions and Authorization** have been filled out for the type of chemical being sent.

☐ ☐ ☐

The **Name, Place & Date, Signature, and Emergency Telephone number** appears at the bottom of the FedEx Airbill.

☐ ☐ ☐

The statement "In accordance with IATA/ICAO" appears in the **Additional Handling Information** box.

# PACKAGING AND SHIPPING OF ENVIRONMENTAL SAMPLES

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Proper Shipping Name	Class or Division	UN or ID No.	Packing Group	Sub Risk	Quantity	Packing Instruction	Authorization
Hydrochloric Acid Solution	8	UN1789	II		1 plastic box x 0.5 L	Y809	LTD QTY
Nitric Acid Solution (with less than 20%)	8	UN2031	II		1 plastic box x 0.5 L	Y807	LTD QTY
Sodium Hydroxide Solution	8	UN1824	II		1 plastic box x 0.5 L	Y809	LTD QTY
Sulfuric Acid Solution	8	UN2796	II		1 plastic box x 0.5 L	Y809	LTD QTY
Hexanes	3	UN1208	II		1 plastic box x 1 L	Y305	LTD QTY

## Sample Cooler Labeling

Yes No N/A

☐ ☐ ☐

The proper shipping name, UN number, and LTD. QTY. appears on the shipping container.

☐ ☐ ☐

The corresponding hazard labels are affixed on the shipping container; the labels are not obscured by tape.

☐ ☐ ☐

The name and address of the shipper and receiver appear on the top and side of the shipping container.

☐ ☐ ☐

The air waybill is attached to the top of the shipping container.

☐ ☐ ☐

Up Arrows have been attached to opposite sides of the shipping container.

☐ ☐ ☐

Packaging tape does not obscure markings or labeling.

**PACKAGING AND SHIPPING OF  
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**APPENDIX B  
SHIPMENT QUALITY ASSURANCE CHECKLIST**

Date: \_\_\_\_\_ Shipper: \_\_\_\_\_ Destination: \_\_\_\_\_

Item(s) Description: \_\_\_\_\_

Radionuclide(s): \_\_\_\_\_

Radiological Survey Results: surface \_\_\_\_\_ mrem/hr 1 meter \_\_\_\_\_

Instrument Used: Mfgr: \_\_\_\_\_ Model: \_\_\_\_\_

S/N: \_\_\_\_\_ Cal Date: \_\_\_\_\_

**LIMITED QUANTITY OR INSTRUMENT AND ARTICLE**

- | Yes | No  |  |
|-----|-----|--|
| ___ | ___ | 1. Strong tight package (package that will not leak material during conditions normally incidental to transportation).   |
| ___ | ___ | 2. Radiation levels at any point on the external surface of package less than or equal to 0.5 mrem/hr.   |
| ___ | ___ | 3. Removable surface contamination less than 20 dpm/100 cm <sup>2</sup> (alpha) and 1000 dpm/100 cm <sup>2</sup> (beta/gamma).   |
| ___ | ___ | 4. Outside inner package bears the marking "Radioactive".  |
| ___ | ___ | 5. Package contains less than 15 grams of <sup>235</sup> U (check yes if <sup>235</sup> U not present).  |
| ___ | ___ | 6. Notice enclosed in or on the package that includes the consignor or consignee and the statement, "This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910." |
| ___ | ___ | 7. Activity less than that specified in 49 CFR 173.425. Permissible package limit:<br>Package Quantity:  |
| ___ | ___ | 8. On all air shipments, the statement, Radioactive Material, excepted package-limited quantity of material shall be noted on the air waybill.   |

Qualified Shipper: \_\_\_\_\_ Signature: \_\_\_\_\_

# FIELD LOGBOOK CONTENT AND CONTROL

SOP 4-1

Revision: 4

Date: June 20, 2001

Page 1 of 5

Prepared: Del Baird

Technical Review: Larry Davidson

QA Review: David O. Johnson

Approved: [Signature]

Issued: [Signature]

6/29/01  
Signature/Date

[Signature]  
Signature/Date

## 1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to set CDM Federal criteria for content entry and form of field logbooks. Field logbooks are an essential tool to document field activities for historical and legal purposes.

## 2.0 BACKGROUND

### 2.1 Definitions

Biota - The flora and fauna of a region.

Magnetic Declination Corrections - Compass adjustments to correct for the angle between magnetic north and geographical meridians.

### 2.2 Discussion

Information recorded in field logbooks includes field team names, observations, data, calculations, date/time, weather, and description of the data collection activity, methods, instruments, and results. Additionally, the logbook may contain deviations from plans and descriptions of wastes, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

## 3.0 RESPONSIBILITIES

**Field Team Leader (FTL)** - The FTL is responsible for ensuring that the format and content of data entries are in accordance with this procedure.

**Site Personnel** - All CDM Federal employees who make entries in field logbooks during onsite activities are required to read this procedure prior to engaging in this activity. The FTL will assign field logbooks to site personnel who will be responsible for their care and maintenance. Site personnel will return field logbooks to the records file at the end of the assignment.

## FIELD LOGBOOK CONTENT AND CONTROL

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### 4.0 REQUIRED EQUIPMENT

- Site-specific plans
- Field notebook
- Indelible black or blue ink pen
- Ruler or similar scale

### 5.0 PROCEDURES

#### 5.1 Preparation

In addition to this SOP, site personnel responsible for maintaining logbooks must be familiar with all procedures applicable to the field activity being performed. These procedures should be consulted as necessary to obtain specific information about equipment and supplies, health and safety, sample collection, packaging, decontamination, and documentation. These procedures should be located at the field office.

Field logbooks shall be bound with lined, consecutively numbered pages. All pages must be numbered prior to initial use of the logbook. Prior to use in the field, each logbook will be marked with a specific document control number issued by the document control administrator, if required by the contract quality implementation plan (QIP). Not all contracts require document control numbers. The following information shall be recorded on the cover of the logbook:

- Field logbook document control number.
- Activity (if the logbook is to be activity-specific) and location.
- Name of CDM Federal contact and phone number(s).
- Start date.
- In specific cases, special logbooks may be required (e.g., waterproof paper for storm water monitoring).

The first few (approximately five) pages of the logbook will be reserved for a table of contents (TOC). Mark the first page with the heading and enter the following:

#### TABLE OF CONTENTS

Date/Description	Page
(Start Date)/Reserved for TOC	1-5

The remaining pages of the table of contents will be designated as such with "TOC" written on the top center of each page.

## FIELD LOGBOOK CONTENT AND CONTROL

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### 5.2 Operation

The following is a list of requirements that must be followed when using a logbook:

- Record work, observations, quantities of materials, calculations, drawings, and related information directly in the logbook. If data collection forms are specified by an activity-specific plan, this information need not be duplicated in the logbook. However, any forms used to record site information must be referenced in the logbook.
- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.
- Do not erase or blot out any entry at any time. Indicate any deletion by a single line through the material to be deleted. Initial and date each deletion. Take care to not obliterate what was written previously.
- Do not remove any pages from the book.

Specific requirements for field logbook entries include:

- Initial and date each page.
- Sign and date the final page of entries for each day.
- Initial and date all changes.
- Multiple authors must sign out the logbook by inserting the following:

Above notes authored by:

- (Sign name)
- (Print name)
- (Date)

- A new author must sign and print his/her name before additional entries are made.
- Draw a diagonal line through the remainder of the final page at the end of the day.
- Record the following information on a daily basis:
  - Date and time
  - Name of individual making entry
  - Names of field team and other persons on site
  - Description of activity being conducted including station or location (i.e., well, boring, sampling location number) if appropriate
  - Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction, and speed) and other pertinent data
  - Level of personal protection to be used
  - Serial numbers of instruments
  - Required calibration information
  - Serial/tracking numbers on documentation (e.g., carrier air bills)

## FIELD LOGBOOK CONTENT AND CONTROL

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Entries into the field logbook shall be preceded with the time (written in military units) of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g., data logger) or on a separate form required by an operating procedure. In these cases, the logbook must reference the automatic data record or form.

At each station where a sample is collected or an observation or measurement made, a detailed description of the location of the station is required. Use a compass (include a reference to magnetic declination corrections), scale, or nearby survey markers, as appropriate. A sketch of station location may be warranted. All maps or sketches made in the logbook should have descriptions of the features shown and a direction indicator. It is preferred that maps and sketches be oriented so that north is toward the top of the page. Maps, sketches, figures, or data that will not fit on a logbook page should be referenced and attached to the logbook to prevent separation.

Other events and observations that should be recorded include:

- Changes in weather that impact field activities.
- Deviations from procedures outlined in any governing documents. Also record the reason for any noted deviation.
- Problems, downtime, or delays.
- Upgrade or downgrade of personal protection equipment.

### 5.3 Post-Operation

To guard against loss of data due to damage or disappearance of logbooks, completed pages shall be periodically photocopied (weekly, at a minimum) and forwarded to the field or project office. Other field records shall be photocopied and submitted regularly and as promptly as possible to the office. When possible, electronic media such as disks and tapes should be copied and forwarded to the project office.

At the conclusion of each activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated, and that corrections were made properly (single lines drawn through incorrect information, then initialed and dated). The completed logbook shall be submitted to the records file.

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### 6.0 RESTRICTIONS/LIMITATIONS

Field logbooks constitute the official record of onsite technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by CDM Federal personnel and their subcontractors. They are documents that may be used in court to indicate dates, personnel, procedures, and techniques employed during site activities. Entries made in these notebooks should be factual, clear, precise, and non-subjective. Field logbooks, and entries within, are not to be utilized for personal use.

### 7.0 REFERENCES

Sandia National Laboratories, *Procedure for Preparing, Sampling and Analysis Plan, Site-Specific Sampling Plan, and Field Operating Procedures*, QA-02-03, Albuquerque Environmental Program Department 3220, Albuquerque, New Mexico, 1991.

Sandia National Laboratories, Division 7723, *Field Operation Procedure for Field Logbook Content and Control*, Environmental Restoration Department, Albuquerque, New Mexico, 1992.



# PHOTOGRAPHIC DOCUMENTATION OF FIELD ACTIVITIES

SOP 4-2

Revision: 5

Date: October 12, 2001

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Prepared: David O. Johnson

Technical Review: Jackie Mosher

QA Review: Doug Updike

Approved: [Signature]

Issued: Rosemary Gustin 10/12/01  
Signature/Date

Signature/Date

## 1.0 OBJECTIVE

The purpose of this standard operating procedure (SOP) is to provide standard guidelines and methods for photographic documentation, which include still and digital photography and videotape recordings of field activities and site features (geologic formations, core sections, lithologic samples, water samples, general site layout, etc.). This document shall provide guidelines designed for use by a professional or amateur photographer. This SOP is intended for circumstances when formal photographic documentation is required. Based on project requirements, it may not be applicable for all photographic activities.

## 2.0 BACKGROUND

### 2.1 Definitions

Photographer – A photographer is the camera operator (professional or amateur) of still photography, including digital photography, or videotape recording whose primary function with regard to this SOP is to produce documentary or data-oriented visual media.

Identifier Component – Identifier components are visual components used within a photograph such as visual slates, reference markers, and pointers.

Standard Reference Marker – A standard reference marker is a reference marker that is used to indicate a feature size in the photograph and is a standard length of measure, such as a ruler, meter stick, etc. In limited instances, if a ruled marker is not available or its use is not feasible, it can be a common object of known size placed within the visual field and used for scale.

Slates – Slates are blank white index cards or paper used to present information pertaining to the subject/ procedure being photographed. Letters and numbers on the slate will be bold and written with black, indelible marking pens.

Arrows and Pointers – Arrows and pointers are markers/pointers used to indicate and/or draw attention to a special feature within the photograph.

Contrasting Backgrounds – Contrasting backgrounds are backdrops used to lay soil samples, cores, or other objects on for clearer viewing and to delineate features.

## **PHOTOGRAPHIC DOCUMENTATION OF FIELD ACTIVITIES**

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Data Recording Camera Back – A data recording camera back is a camera attachment or built-in feature that will record, at the very least, frame numbers and dates directly on the film.

### **2.2 Discussion**

Photographs and videotape recordings made during field investigations are used as an aid in documenting and describing site features, sample collection activities, equipment used, and possible lithologic interpretation. This SOP is designed to illustrate the format and desired placement of identifier components, such as visual slates, standard reference markers, and pointers. These items shall become an integral part of the "visual media" that, for the purpose of this document, shall encompass still photographs, digital photographs, and videotape recordings (or video footage). The use of a photographic logbook and standardized entry procedures are also outlined. These procedures and guidelines will minimize potential ambiguities that may arise when viewing the visual media and ensure the representative nature of the photographic documentation.

### **2.3 Associated Procedures**

- CDM Federal SOP 4-1, Field Logbook Content and Control

## **3.0 RESPONSIBILITIES**

**Field Team Leader (FTL)** – The FTL is responsible for ensuring that the format and content of photographic documentation are in accordance with this procedure. The FTL is responsible for directing the photographer to specific situations, site features, or operations that the photographer will be responsible for documenting.

**Photographer** – The photographer shall seek direction from the FTL and regularly discuss the visual documentation requirements and schedule. The photographer is responsible for maintaining a logbook per Sections 5.1, 5.2.4, and 5.3.1 of this SOP.

## **4.0 REQUIRED EQUIPMENT**

The following is a general list of equipment that may be used:

- 35mm camera or disposable single use camera (35mm or panoramic use)
- Digital camera
- Video camera
- Logbook
- Indelible black or blue ink pen
- Standard reference markers
- Slates
- Arrows or pointers

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- Contrasting backgrounds
- Medium speed, or multi purpose fine-grain, color, 35 mm, negative film or slide film (project dependent)
- Data recording camera back (if available)
- Storage medium for digital camera

### **5.0 PROCEDURES**

#### **5.1 Documentation**

A commercially available, bound logbook will be used to log and document photographic activities. Review the CDM Federal SOP 4-1 (Field Logbook Content and Control) and prepare all supplies needed for logbook entries.

Note: A separate photographic logbook is not required. A portion of the field logbook may be designated as the photographic log and documentation section.

##### **5.1.1 Field - Health and Safety Considerations**

There are no hazards that an individual will be exposed to specific to photographic documentation. However, site-specific hazards may arise depending on location or operation. Personal protective equipment used in this operation will be site-specific and dictated through requirements set by the site safety officer, site health and safety plan, and/or prescribed by the CDM Federal Corporate Health and Safety Program. The photographer should contact the site safety officer for health and safety orientation prior to commencing field activities. The site health and safety plan must be read prior to entry to the site, and all individuals must sign the appropriate acknowledgement that this has been done.

The photographer should be aware of any potential physical hazards while photographing the subject (e.g., low overhead hazard, edge of excavation).

### **5.2 OPERATION**

#### **5.2.1 General Photographic Activities in the Field**

The following sections provide general guidelines that should be followed to visually document field activities and site features using still/digital cameras and video equipment. Listed below are general suggestions that the photographer should consider when performing activities under this SOP:

- The photographer should be prepared to make a variety of shots, from closeup to wide-angle. Many shots will be repetitive in nature or format especially closeup site feature photographs. Consideration should therefore be given to designing a system or technique that will provide a reliable repetition of performance.

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- All still film photographs should be made using a medium speed, or multi purpose fine-grain, color negative film in the 35 mm format unless otherwise directed by the FTL.
- It is suggested that Kodak brand "Ektapress Gold Deluxe" film or equivalent be used as the standard film for the still photography requirements of the field activities. This film is stable at room temperature after exposure and will better survive the time lag between exposure and processing. It is suggested that film speed ASA 100 should be used for outdoor photographs in bright sunlight, ASA 200 film should be used in cloudy conditions, and ASA 400 film should be used indoors or for very low-light outdoor photographs.
- No preference of videotape brand or digital storage medium is specified and is left to the discretion of the photographer.
- The lighting for sample and feature photography should be oriented toward a flat condition with little or no shadow. If the ambient lighting conditions are inadequate, the photographer should be prepared to augment the light (perhaps with reflectors or electronic flash) to maintain the desired visual effect.
- Digital cameras have multiple photographic quality settings. A camera that obtains a higher resolution (quality) has a higher number of pixels and will store a fewer number of photographs per digital storage medium.

### 5.2.2 General Guidelines for Still Photography

#### Slate Information

When directed by the FTL, each new roll of film or digital storage medium shall contain upon the first usable frame (for film) a slate with consecutively assigned control numbers (a consecutive, unique number that is assigned by the photographer as in sample numbers).

#### Caption Information

All still photographs will have a full caption permanently attached to the back or permanently attached to a photo log sheet. The caption should contain the following information (digital photographs should have a caption added after the photographs are downloaded):

- Film roll control number (if required) and photograph sequence number
- Date and time
- Description of activity/item shown
- Direction (if applicable)
- Photographer

When directed by the FTL, a standard reference marker should be used in all documentary visual media. While the standard reference marker will predominantly be used in closeup feature documentation, inclusion in all scenes should be considered.

Digital media should be downloaded at least once each day.

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### **Closeup and Feature Photography**

When directed by the FTL, closeup photographs should include a standard reference marker of appropriate size as an indication of the feature size and contain a slate marked with the site name and any identifying label, such as a well number or core depth, that clearly communicates to the viewer the specific feature being photographed.

Feature samples, core pieces, and other lithologic media should be photographed as soon as possible after they have been removed from their in situ locations. This enables a more accurate record of their initial condition and color. When directed by the FTL, include a standard reference color strip (color chart such as Munsell Soil Color Chart or that available from Eastman Kodak Co.) within the scene. This is to be included for the benefit of the viewer of the photographic document and serves as a reference aid to the viewer for formal lithologic observations and interpretations.

### **Site Photography**

Site photography, in general, will consist predominantly of medium and wide-angle shots. A standard reference marker should be placed adjacent to the feature or, when this is not possible, within the same focal plane.

While it is encouraged that a standard reference marker and caption/slate be included in the scene, it is understood that situations will arise that preclude their inclusion within the scene. This will be especially true of wide-angle shots. In such a case, the film/tape control number shall be entered in the photographic logbook along with the frame number and all other information pertinent to the scene.

### **Panoramic**

In situations where a wide-angle lens does not provide sufficient subject detail, a single-use disposable panoramic camera is recommended. If this type of camera is not available, a panoramic series of two or three photos would be appropriate. Panoramas can provide greater detail while covering a wide subject, such as an overall shot of a site.

To shoot a panoramic series using a standard 35mm or digital camera, the following procedure is recommended.

- Use a stable surface or tripod to support the camera.
- Allow a 20 to 30 percent overlap while maintaining a uniform horizon.
- Complete 2 to 3 photos per series.

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### **5.2.3 General Photographic Documentation Using Video Cameras**

As a reminder, it is not within the scope of this document to set appropriate guidelines for presentation or "show" videotape recording. The following guidelines are set for documentary videotape recordings only and should be implemented at the discretion of the FTL.

Documentary videotape recordings of field activities may include an audio slate for all scenes. At the beginning of each video session, an announcer will recite the following information: date, time (in military units), photographer, site ID number, and site location. This oral account may include any additional information clarifying the subject matter being recorded.

A standard reference marker may be used when taking closeup shots of site features with a video camera. The scene may also include a caption/slate. It should be placed adjacent and parallel to the feature being photographed.

It is recommended that a standard reference marker and caption/slate be included in all scenes. The caption information is vital to the value of the documentary visual media and should be included. If it is not included within the scene, it should be placed before the scene.

Original videotape recordings will not be edited. This will maintain the integrity of the information contained on the videotape. If editing is desired, a working copy of the original videotape recording can be made.

### **5.2.4 Photographic Documentation**

Photographic activities must be documented in a photographic logbook or in a section of the field logbook. The photographer will be responsible for making proper entries.

In addition to following the technical standards for logbook entry as referenced in CDM Federal SOP 4-1, the following information should be maintained in the appropriate logbook:

- Photographer name.
- If required, an entry shall be made for each new roll/tape control number assigned.
- Sequential tracking number for each photograph taken (for digital cameras, the camera-generated number may be used).
- Date and time (military time).
- Location.
- A description of the activity/item photographed.
- If needed, a description of the general setup, including approximate distance between the camera and the subject, may be recorded in the logbook.
- Record as much other information as possible to assist in the identification of the photographic document.

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### **5.3 Post Operation**

All film will be sent for development and printing to a photographic laboratory (to be determined by the photographer). The photographer will be responsible for arranging transport of the film from the field to the photographic laboratory. The photographer shall also be responsible for arranging delivery of the negatives and photographs, digital storage medium, or videotape to the project management representative.

#### **5.3.1 Documentation**

At the end of each day's photographic session, the photographer(s) will ensure that the appropriate logbook has been completely filled out and maintained as outlined in CDM Federal SOP 4-1.

#### **5.3.2 Archive Procedures**

1. Photographs and the associated set of negatives, digital media, and original unedited documentary videotape recordings will be submitted to the project files and handled according to contract records requirements. The FTL will ensure their proper distribution.
2. Completed pages of the appropriate logbook will be copied weekly and submitted to the project files.

### **6.0 RESTRICTIONS/LIMITATIONS**

This document is designed to provide a set of guidelines for the field amateur or professional photographer to ensure that an effective and standardized program of visual documentation is maintained.

It is not within the scope of this document to provide instruction in photographic procedures, nor is it within the scope of this document to set guidelines for presentation or "show" photography.

The procedures outlined herein are general by nature. The FTL is responsible for specific operational activity or procedure. Questions concerning specific procedures or requirements should be directed to the FTL.

**NOTE:** Some sites do not permit photographic documentation. Check with the site contact for any restrictions.

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### 7.0 REFERENCES

U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans*, EM 200-1-3, February 2001, Appendix F.

U.S. Environmental Protection Agency, Region IV, *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, Athens, Georgia, May 1996.

U.S. Environmental Protection Agency, National Enforcement Investigations Center, *Multi-Media Investigation Manual*, EPA-330/9-89-003-R, Revised March 1992, p. 85.



# FIELD EQUIPMENT DECONTAMINATION AT NONRADIOACTIVE SITES

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Prepared: Steven Fundingsland

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Issued: [Signature] 12/10/02  
Signature/Date

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## 1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to describe the procedures required for decontamination of field equipment.

## 2.0 BACKGROUND

### 2.1 Definitions

Clean - Free of visible contamination and when decontamination has been completed in accordance with this SOP.

Cross-Contamination - The transfer of contaminants through equipment or personnel from the contamination source to less contaminated or non-contaminated samples or areas.

Decontamination - The process of rinsing or otherwise cleaning the surfaces of equipment to rid them of contaminants and to minimize the potential for cross contamination of samples or exposure of personnel.

### 2.2 Discussion

Decontamination of field equipment is necessary to ensure acceptable quality of samples by preventing cross contamination. Further, decontamination reduces health hazards and prevents the spread of contaminants off-site.

## 3.0 RESPONSIBILITIES

**Field Team Leader** - The Field Team Leader (FTL) ensures that field personnel are trained in the performance of this procedure and that decontamination is conducted in accordance with this procedure. The FTL may also be required to collect and document rinsate samples to provide quantitative verification that these procedures have been correctly implemented.

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### **4.0 REQUIRED EQUIPMENT**

- Stiff-bristle scrub brushes
- Plastic buckets and troughs
- Laboratory-grade detergent, low phosphate (Alconox™, Liquinox™ or similar)
- Nalgene or Teflon Sprayers or wash bottles or 2- to 5-gallon, manual-pump sprayer (pump sprayer material must be compatible with the solution used)
- Plastic sheeting
- Disposable wipes, rags or paper towels
- Potable water and/or de-ionized water of American Society for Testing and Materials (ASTM) Type II or better, as defined by ASTM Standard Specification for Reagent Water, Standard D 1193-77 (re-approved 1983)\*
- Gloves, safety glasses, and other protective clothing as specified in the site-specific health and safety plan
- High-pressure pump with soap dispenser or steam-spray unit (for large equipment only)
- Appropriate decontamination solutions pesticide grade or better and traceable to a source (e.g. 10% and/or 1% nitric acid (HNO<sub>3</sub>), acetone, methanol, isopropanol, hexane)
- Tools for equipment assembly and disassembly (as required)
- 55-gallon drums or tanks (as required)
- Pallets for drums or tanks holding decontamination water (as required)

\* Potable water may be required to be tested for contaminants before use. Check field plan for requirements. ASTM Type II water will include a certificate of quality.

### **5.0 PROCEDURES**

All reusable equipment (non-dedicated) used to collect, handle, or measure samples will be decontaminated before coming into contact with any sample. Decontamination of equipment will occur either at a central decontamination station or at portable decontamination stations set up at the sampling location, drill site, or monitoring well location. The centrally-located decontamination station will include an appropriately sized bermed and lined area on which equipment decontamination will occur and shall be equipped with a collection system and storage vessels. In certain circumstances, berming is not required when small quantities of water are being generated and for some short duration field activities (i.e., pre-remedial sampling). Equipment should be transported to the decontamination station in a manner to prevent cross-contamination of equipment and/or area. Precautions taken may include enclosing augers in plastic wrap while being transported on a flatbed truck.

The decontamination area will be constructed so that contaminated water is either collected directly into appropriate containers (5-gallon buckets or steel wash tubs) or within the berms of the

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decontamination area which then drains into a collection system. Water from the collection system will be transferred into 55-gallon drums or portable tanks for storage. Typically, decontamination water will be staged until sampling results or waste characterization results are obtained and evaluated and the proper disposition of the waste is determined. The exact procedure for decontamination waste disposal should be discussed in the field plan. Also, decontamination fluids, such as solvents, may need to be segregated from other investigation-derived wastes.

All items that will come into contact with potentially contaminated media will be decontaminated before use and between sampling and/or drilling locations. If decontaminated items are not immediately used, they will be covered either with clean plastic or aluminum foil depending on the size of the item. All decontamination procedures for the equipment being used are as follows:

### General Guidelines

- Potable and de-ionized water should be free of all contaminants of concern. Following the field plan, analytical data from the water source may be required. If required, either existing analytical data from the water source supplier (i.e., municipality, bottled water company, de-ionized water producer) may be obtained or chemical testing may be performed on the selected source.
- Soap will be a low phosphate detergent.
- Sampling equipment that has come into contact with oil and grease will be cleaned with methanol or other approved alternative to remove the oily material. This may be followed by a hexane rinse and then another methanol rinse. Regulatory or client requirements regarding solvent use will be stated in the field plan.
- All solvents will be pesticide grade or better and traceable to a source. The corresponding lot numbers will be recorded in the appropriate logbook.
- Decontaminated equipment will be allowed to air dry before being used.
- Documentation for all cleaning will be recorded in the appropriate logbook.
- Gloves, boots, safety glasses, and any other personnel protective clothing and equipment will be used as specified in the site-specific health and safety plan.

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### **5.1 Heavy Equipment Decontamination**

Heavy equipment includes drilling rigs and backhoes. Follow these steps when decontaminating this equipment:

1. Establish a decontamination area with berms that is large enough to fully contain the equipment to be cleaned. If available, an existing wash pad or appropriate paved and bermed area may be utilized; otherwise, use one or more layers of heavy plastic sheeting to cover the ground surface and berms. All decontamination pads should be upwind of the area under investigation.
2. With the rig in place, spray areas (rear of rig or backhoe) exposed to contaminated soils using a hot water high-pressure sprayer. Be sure to spray down all surfaces, including the undercarriage.
3. Use brushes, low phosphate detergent and potable water to remove dirt whenever necessary.
4. Remove equipment from the decontamination pad and allow it to air dry before returning it to the work site.
5. Record equipment type, date, time, and method of decontamination in the appropriate logbook.
6. After decontamination activities are completed, collect all contaminated wastewater, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal as detailed in the field plan. Liquids and solids must be drummed separately.

### **5.2 Downhole Equipment Decontamination**

Downhole equipment decontamination includes hollow-stem augers, drill pipes, casings, screens, etc. Follow these steps when decontaminating this equipment:

1. Set up a centralized decontamination area, if possible. This area should be set up to collect contaminated rinse waters and to minimize the spread of airborne spray.
2. Set up a "clean" area upwind of the decontamination area to receive cleaned equipment for air-drying. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or other surfaces on which decontaminated equipment is to be placed. All decontamination pads should be upwind of any areas under investigation.

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3. Place the object to be cleaned on aluminum foil or plastic-covered wooden sawhorses or other supports.
4. Using low phosphate detergent and potable water in the hot water high-pressure sprayer (or steam unit), spray the contaminated equipment. Aim downward to avoid spraying outside the decontamination area. Be sure to spray inside corners and gaps especially well. Use a brush, if necessary, to dislodge dirt.
5. If using soapy water, rinse the equipment using clean, potable water. If using hot water, the rinse step is not necessary if the hot water does not contain a detergent. If the hot water contains a detergent, this final clean water rinse is required.
6. Using the manual-pump sprayer, rinse the equipment thoroughly with de-ionized water (ASTM Type II or better).
7. Remove the equipment from the decontamination area and place in a clean area upwind to air dry.
8. Record equipment type, date, time, and method of decontamination in the appropriate logbook.
9. After decontamination activities are completed, collect all contaminated wastewaters, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal. Liquids and solids must be drummed separately.

### **5.3 Sampling Equipment Decontamination**

Sampling equipment includes split spoons, spatulas, and bowls used for sample homogenization that directly contact sample media. Follow these steps when decontaminating this equipment:

1. Set up a decontamination line on plastic sheeting. The decontamination line should progress from "dirty" to "clean" and have an area located upwind for drying decontaminated equipment. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or the surfaces on which decontaminated equipment is to be placed for drying.
2. Before washing, disassemble any items that might trap contaminants internally. Do not reassemble these items until decontamination and air-drying are complete. Wash items thoroughly in a bucket of low phosphate detergent and potable water. Use a stiff-bristle brush to dislodge any gross contamination (soil or debris).

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3. Rinse the item in potable water. Rinse water should be replaced as needed, generally when cloudy.
4. Using a hand sprayer, wash bottles, or manual-pump sprayer, rinse the item with de-ionized water (ASTM Type II or better).
5. If sampling for metal analytes, rinse the item with 10% nitric acid (for stainless steel, glass, plastic, and Teflon), or 1% nitric acid (for items made of low-carbon steel) followed by a de-ionized water (ASTM Type II or better) rinse.

**NOTE:** Care should be taken not to get nitric acid on skin or clothing. This step should not be used unless required by sampling needs as dictated in the field plan.

**CAUTION:** Do not allow nitric acid to contact methanol or hexane. Contain nitric acid waste separate from organic solvents.

6. If sampling for organic analytes, rinse the item with methanol or approved organic solvent.
7. If required by the field plan, when sampling for polar organic compounds such as pesticides, polychlorinated biphenyls (PCBs), and fuels, rinse the item with hexane or approved alternatives, followed by a second methanol rinse.
8. Thoroughly rinse the item with de-ionized water (ASTM Type II or better).
9. Allow the item to air dry completely.
10. After drying, reassemble parts as required and wrap the item in clean plastic wrap or in aluminum foil, shiny side out.
11. Record equipment type, date, time, and method of decontamination in the appropriate logbook.
12. After decontamination activities are completed, collect all contaminated waters, used solvents and acids, plastic sheeting, and disposable gloves, boots, and clothing. Place contaminated items in properly labeled drums for disposal. Liquids and solids must be drummed separately. (Refer to site-specific plans for labeling and waste management requirements).

### 5.4 Pump Decontamination

Follow the manufacturer's recommendation for specified pump decontamination procedures. At a minimum follow these steps when decontaminating pumps:

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1. Set up the decontamination area and separate "clean" storage area using plastic sheeting to cover the ground, tables, and other surfaces. Set up three 55-gallon drums and one or more containers of ASTM Type II water (or as specified in the field plan). One drum shall contain dilute (non-foaming) soapy water, the second drum shall contain potable water, and the third drum shall be empty to receive waste water.
2. The pump should be set up in the same configuration as for sampling. Submerge the pump intake (or the pump, if submersible) and all downhole-wetted parts (tubing, piping, foot valve) in the soapy water of the first drum. Place the discharge outlet in the wastewater drum above the level of the wastewater. Pump soapy water through the pump assembly until it discharges to the waste drum. Scrub the outside of the pump and other wetted parts with a metal brush.
3. Move the pump assembly to the potable water drum while leaving discharge outlet in the waste drum. All downhole-wetted parts must be immersed in the potable water rinse. Pump potable water through the pump assembly until it runs clear.
4. Move the pump intake to the ASTM Type II water can. Pump the ASTM Type II water through the pump assembly. Pump the volume of water through the pump specified in the field plan. Usually, three pump-and-line-assembly volumes will be required.
5. Decontaminate the discharge outlet by hand following the steps outlined in Section 5.3.
6. Remove the decontaminated pump assembly to the "clean" area and allow it to air dry upwind of the decontamination area. Intake and outlet orifices should be covered with aluminum foil to prevent the entry of airborne contaminants and particles.
7. Record the equipment type, serial number, date, time, and method of decontamination in the appropriate logbook.

### **5.5 Instrument Probe Decontamination**

Instrument probes used for field measurements such as pH meters, conductivity meters, etc. will be decontaminated between samples and after use with ASTM Type II, or better, water.

### **5.6 Waste Disposal**

Refer to site-specific plans for waste disposal requirements. The following are guidelines for disposing of wastes:

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1. All wash water, rinse water, and decontamination solutions that have come in contact with contaminated equipment are to be handled, packaged, labeled, marked, stored, and disposed of as investigation-derived waste.
2. Small quantities of decontamination solutions may be allowed to evaporate to dryness.
3. If large quantities of used decontamination solutions will be generated, each type of waste should be separated in separate containers. This may permit the disposal of wash water and rinse water onsite or in a sanitary sewage treatment plant rather than as a hazardous waste. If an industrial wastewater treatment plant is available onsite, the disposal of acid solutions and solvent-water solutions may be permitted.
4. Unless otherwise required, plastic sheeting and disposable protective clothing may be treated as solid, non-hazardous waste.
5. Waste liquids should be sampled, analyzed for contaminants of concern in accordance with disposal regulations, and disposed of accordingly.

### 6.0 RESTRICTIONS/LIMITATIONS

Nitric acid and polar solvent rinses are necessary only when sampling for metals or organics respectively. These steps should not be used, unless required, because of the potential for acid burns and ignitability hazards.

If the field equipment is not thoroughly rinsed and allowed to completely air dry before use, volatile organic residue, which interferes with the analysis, may be detected in the samples. The occurrence of residual organic solvents is often dependent on the time of year sampling is conducted. In the summer, volatilization is rapid, and in the winter, volatilization is slow. Check with your EPA region, state, and client for approved decontamination solvents.

### 7.0 REFERENCES

Department of Energy, Hazardous Waste Remedial Actions Program, *Standard Operating Procedures For Site Characterization*, DOE/HWP-100/R1, September 1996.

Department of Energy, Hazardous Waste Remedial Actions Program, *Quality Control Requirements For Field Methods*, DOE/HWP-69/R2, September 1996.

American Society for Testing and Materials, *Standard Practice for Decontamination of Field Equipment at Nonradioactive Waste Sites*, ASTM D5088-90, June 29, 1990.



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U.S. Environmental Protection Agency, Region II, *"CERCLA" Quality Assurance Manual*, Revision 1, 1989.

U.S. Environmental Protection Agency, Region IV, *Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual*, 1986.

U.S. Environmental Protection Agency, *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001.1, 1987.



## ASBESTOS SAMPLING

SOP#: 2015  
DATE: 11/17/94  
REV. #: 0.0

### 1.0 SCOPE AND APPLICATION

Asbestos has been used in many commercial products including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, and roofing asphalts. These products and others may be found at hazardous waste sites hanging on overhead pipes, contained in drums, abandoned in piles, or as part of a structure. Asbestos tailing piles from mining operations can also be a source of ambient asbestos fibers. Asbestos is a known carcinogen and requires air sampling to assess airborne exposure to human health. This Standard Operating Procedure (SOP) provides procedures for asbestos air sampling by drawing a known volume of air through a mixed cellulose ester (MCE) filter. The filter is then sent to a laboratory for analysis. The U.S. Environmental Protection Agency/Environmental Response Team (U.S. EPA/ERT) uses one of four analytical methods for determining asbestos in air. These include: U.S. EPA's Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air for Transmission Electron Microscopy (TEM)<sup>(1)</sup>; U.S. EPA's Modified Yamate Method for TEM<sup>(2)</sup>; National Institute for Occupational Safety and Health (NIOSH) Method 7402 (direct method only) for TEM; and NIOSH Method 7400 for Phase Contrast Microscopy (PCM)<sup>(3)</sup>. Each method has specific sampling and analytical requirements (i.e., sample volume and flow rate) for determining asbestos in air.

The U.S. EPA/ERT typically follows procedures outlined in the TEM methods for determining mineralogical types of asbestos in air and for distinguishing asbestos from non-asbestos minerals. The Phase Contrast Microscopy (PCM) method is used by U.S. EPA/ERT as a screening tool since it is less costly than TEM. PCM cannot distinguish asbestos from non-asbestos fibers, therefore the TEM method may be necessary to confirm analytical results. For example, if an action level for the presence of fibers has been set and PCM analysis indicates that the action level has been exceeded, then

TEM analysis can be used to quantify and identify asbestos structures through examination of their morphology, crystal structures (through electron diffraction), and elemental composition (through energy dispersive X-ray analysis). In this instance, samples should be collected for both analyses in side by side sampling trains (some laboratories are able to perform PCM and TEM analysis from the same filter). The Superfund method is designed specifically to provide results suitable for supporting risk assessments at Superfund sites; it is applicable to a wide range of ambient air situations at hazardous waste sites. U.S. EPA's Modified Yamate Method for TEM is also used for ambient air sampling due to high volume requirements. The PCM and TEM NIOSH analytical methods require lower sample volumes and are typically used indoors; however, ERT will increase the volume requirement for outdoor application.

Other Regulations pertaining to asbestos have been promulgated by U.S. EPA and OSHA. U.S. EPA's National Emission Standards for Hazardous Air Pollutants (NESHAP) regulates asbestos-containing waste materials. NESHAP establishes management practices and standards for the handling of asbestos and emissions from waste disposal operations (40 CFR Part 61, Subparts A and M). U.S. EPA's 40 CFR 763 (July 1, 1987)<sup>(4)</sup> and its addendum 40 CFR 763 (October 30, 1987)<sup>(4)</sup> provide comprehensive rules for the asbestos abatement industry. State and local regulations on these issues vary and may be more stringent than federal requirements. The OSHA regulations in 29 CFR 1910.1001 and 29 CFR 1926.58 specify work practices and safety equipment such as respiratory protection and protective clothing when handling asbestos. The OSHA standard for an 8-hour, time-weighted average (TWA) is 0.2 fibers/cubic centimeters of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length  $>5 \mu\text{m}$ <sup>(5,6)</sup>. An action level of 0.1 fiber/cc (one-half the OSHA standard) is the level U.S. EPA has established in which employers must initiate such activities as air monitoring, employee training, and

medical surveillance <sup>(3,6)</sup>.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

## 2.0 METHOD SUMMARY

Prior to sampling, the site should be characterized by identifying on-site as well as off-site sources of airborne asbestos. The array of sampling locations and the schedule for sample collection, is critical to the success of an investigation. Generally, sampling strategies to characterize a single point source are fairly straightforward, while multiple point sources and area sources increase the complexity of the sampling strategy. It is not within the scope of this SOP to provide a generic asbestos air sampling plan. Experience, objectives, and site characteristics will dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to distinguish spatial trends in airborne asbestos concentrations. Sampling schedules should be fashioned to establish temporal trends. The sampling strategy typically requires that the concentration of asbestos at the source (worst case) or area of concern (downwind), crosswind, as well as background (upwind) contributions be quantified. See Table 1 (Appendix A) for U.S. EPA/ERT recommended sampling set up for ambient air. Indoor asbestos sampling requires a different type of strategy which is identified in Table 2 (Appendix A). It is important to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine other sources.

Much information can be derived from each analytical method previously mentioned. Each analytical method has specific sampling requirements and produce results which may or may not be applicable to a specific sampling effort. The site sampling

objectives should be carefully identified so as to select the most appropriate analytical method. Additionally, some preparation (i.e., lot blanks results) prior to site sampling may be required, these requirements are specified in the analytical methods.

## 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

### 3.1 Sample Preservation

No preservation is required for asbestos samples.

### 3.2 Sample Handling, Container and Storage Procedures

1. Place a sample label on the cassette indicating a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up fibers. The original cassette box is used to hold the samples.
2. Wrap the cassette individually in a plastic sample bag. Each bag should be marked indicating sample identification number, total volume, and date.
3. The wrapped sampling cassettes should be placed upright in a rigid container so that the cassette cap is on top and cassette base is on bottom. Use enough packing material to prevent jostling or damage. Do not use vermiculite as packing material for samples. If possible, hand carry to lab.
4. Provide appropriate documentation with samples (i.e., chain of custody and requested analytical methodology).

## 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) which could result in filter destruction due to (a) failure of its physical support under force from the increased pressure drop; (b) leakage of air around the filter mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impact velocities.

## 4.1 U.S. EPA's Superfund Method

### 4.1.1 Direct-transfer TEM Specimen Preparation Methods

Direct-Transfer TEM specimen preparation methods have the following significant interferences:

- The achievable detection limit is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.
- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.
- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting will not be possible.

### 4.1.2 Indirect TEM Specimen Preparation Methods

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.
- There is increased opportunity for fiber loss or introduction of extraneous contamination.
- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate

during the preparation, resulting in an increase in the numbers of structures counted.

## 4.2 U.S. EPA's Modified Yamate Method for TEM

High concentrations of background dust interfere with fiber identification.

## 4.3 NIOSH Method for TEM

Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification.

## 4.4 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting criteria are counted as total asbestos fibers. Fiber less than 0.25  $\mu\text{m}$  in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

## 5.0 EQUIPMENT/MATERIALS

### 5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled.

The lower flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters/minute (cc/min) to 4 L/min. These pumps are usually battery powered. High flow pumps are utilized when flow rates between 2 L/min to 20 L/min are required. High flow pumps are used for short sampling periods so as to obtain the desired sample volume. High flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several

electrical extension cords may be required if sampling locations are remote.

The recommended volume for the Superfund method (Phase I) requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power so that 2 lead/acid batteries should provide sufficient power to collect a full sample. The use of line voltage, where available, eliminates the difficulties associated with transporting stored electrical energy.

A stand should be used to hold the filter cassette at the desired height for sampling and the filter cassette shall be isolated from the vibrations of the pump.

## 5.2 Filter Cassette

The cassettes are purchased with the required filters in position, or can be assembled in a laminar flow hood or clean area. When the filters are in position, a shrink cellulose band or adhesive tape should be applied to cassette joints to prevent air leakage.

### 5.2.1 TEM Cassette Requirements

Commercially available field monitors, comprising 25 mm diameter three-piece cassettes, with conductive extension cowls shall be used for sample collection. The cassette must be new and not previously used. The cassette shall be loaded with an MCE filter of pore size 0.45  $\mu\text{m}$ , and supplied from a lot number which has been qualified as low background for asbestos determination. The cowl should be constructed of electrically conducting material to minimize electrostatic effects. The filter shall be backed by a 5  $\mu\text{m}$  pore size MCE filter (Figure 1, Appendix B).

### 5.2.2 PCM Cassette Requirements

NIOSH Method 7400, PCM involves using a 0.8 to 1.2  $\mu\text{m}$  mixed cellulose ester membrane, 25 mm diameter, 50 mm conductive cowl on cassette (Figure 2, Appendix B). Some labs are able to perform PCM and TEM analysis on the same filter; however, this should be discussed with the laboratory prior to sampling.

## 5.3 Other Equipment

- Inert tubing with glass cyclone and hose barb
- Whirlbags (plastic bags) for cassettes

- Tools - small screw drivers
- Container - to keep samples upright
- Generator or electrical outlet (may not be required)
- Extension cords (may not be required)
- Multiple plug outlet
- Sample labels
- Air data sheets
- Chain of Custody records

## 6.0 REAGENTS

Reagents are not required for the preservation of asbestos samples.

## 7.0 PROCEDURES

### 7.1 Air Volumes and Flow Rates

Sampling volumes are determined on the basis of how many fibers need to be collected for reliable measurements. Therefore, one must estimate how many airborne fibers may be in the sampling location.

Since the concentration of airborne aerosol contaminants will have some effect on the sample, the following is a suggested criteria to assist in selecting a flow rate based on real-time aerosol monitor (RAM) readings in milligrams/cubic meter ( $\text{mg}/\text{m}^3$ ).

	Concentration	Flow Rate
• Low RAM readings:	$<6.0 \text{ mg}/\text{m}^3$	11-15 L/min
• Medium RAM readings:	$>6.0 \text{ mg}/\text{m}^3$	7.5 L/min
• High RAM readings:	$>10. \text{ mg}/\text{m}^3$	2.5 L/min

In practice, pumps that are available for environmental sampling at remote locations operate under a maximum load of approximately 12 L/min.

#### 7.1.1 U.S. EPA's Superfund Method

The Superfund Method incorporates an indirect preparation procedure to provide flexibility in the amount of deposit that can be tolerated on the sample filter and to allow for the selective concentration of asbestos prior to analysis. To minimize contributions to background contamination from asbestos present in the plastic matrices of membrane filters while allowing for sufficient quantities of asbestos to be collected, this method also requires the collection of a larger volume of air per unit area of filter than has traditionally been collected

for asbestos analysis. Due to the need to collect large volumes of air, higher sampling flow rates are recommended in this method than have generally been employed for asbestos sampling in the past. As an alternative, samples may be collected over longer time intervals. However, this restricts the flexibility required to allow samples to be collected while uniform meteorological conditions prevail.

The sampling rate and the period of sampling should be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters (15,000 L) shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method. One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed. It has been found that the volume cannot normally exceed 5 cubic meters (5000 L) in an urban or agricultural area, and 10 cubic meters (10,000 L) in a rural area for samples collected on a 25 mm filter and prepared by a direct-transfer technique.

An upper limit to the range of acceptable flow rates for this method is 15 L/min. At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options. Air volumes collected on Phase 1 samples are maximized (<16 L/min). Air volumes collected on Phase 2 samples are limited to provide optimum loading for filters to be prepared by a direct-transfer procedure.

### 7.1.2 U.S. EPA's Modified Yamamoto Method for TEM

U.S. EPA's TEM method requires a minimum volume

of 560 L and a maximum volume of 3,800 L in order to obtain an analytical sensitivity of 0.005 structures/cc. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25 mm (385 sq mm) and 37 mm (855 sq mm) differ.

### 7.1.3 NIOSH Method for TEM and PCM

The minimum recommended volume for TEM and PCM is 400 L at 0.1 fiber/cc. Sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (700 to 2800 L) is appropriate in non-dusty atmospheres containing 0.1 fiber/cc. Dusty atmospheres i.e., areas with high levels of asbestos, require smaller sample volumes (<400 L) to obtain countable samples.

In such cases, take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If > 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration. Do not exceed 0.5 mg total dust loading on the filter.

## 7.2 Calibration Procedures

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette cannot be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette."

A rotameter can be used provided it has been recently precalibrated with a primary calibrator. Three separate constant flow calibration readings should be obtained both before sampling and after sampling. Should the flow rate change by more than 5% during the sampling period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. The sampling pump used shall provide a non-fluctuating air-flow through the filter, and shall maintain the initial volume flow-rate to within  $\pm 10\%$  throughout the sampling period. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. Flexible tubing is used to connect the filter cassette to the sampling pump. Sampling pumps can be calibrated prior to coming on-site so that time is saved when performing on-site calibration.

#### 7.2.1 Calibrating a Personal Sampling Pump with an Electronic Calibrator

1. See Manufacturer's manual for operational instructions.
2. Set up the calibration train as shown in (Figure 3, Appendix B) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.
3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.
4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.
5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. Perform the calibration three times until the desired flow rate of  $\pm 5\%$  is attained.

#### 7.2.2 Calibrating a Rotameter with an Electronic Calibrator

1. See manufacturer's manual for operational instructions.
2. Set up the calibration train as shown in (Figure 4, Appendix B) using a sampling pump, rotameter, and electronic calibrator.
3. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.
4. Turn the electronic calibrator and sampling pump on.
5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.
6. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.
7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range. Readings can also be calibrated for 10 cm<sup>3</sup> increments for Low Flow rotameters, 50.0 cm<sup>3</sup> increments for medium flow rotameters and 1 liter increments for high flow rotameters.
8. Perform the calibration three times until the desired flow rate of  $\pm 5\%$  is attained. Once on site, a secondary calibrator, i.e., rotameter may be used to calibrate sampling pumps.

#### 7.2.3 Calibrating a Personal Sampling Pump with a Rotameter

1. See manufacturer's manual for Rotameter's Operational Instructions.

2. Set up the calibration train as shown in (Figure 5, Appendix B) using a rotameter, sampling pump, and a representative sampling cassette.
3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 ft) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.
4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.
5. Turn the sampling pump on.
6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the precalibrated flow rate value. A sticker on the rotameter should indicate this value.
7. A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

### 7.3. Meteorology

It is recommended that a meteorological station be established. If possible, sample after two to three days of dry weather and when the wind conditions are at 10 mph or greater. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind direction is particularly important when monitoring for asbestos downwind from a fixed source.

## 7.4 Ambient Sampling Procedures

### 7.4.1 Pre-site Sampling Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.
2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).

3. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety plan.
4. Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.
5. After calibrating the sampling pump, mobilize to the sampling location.

### 7.4.2 Site Sampling

1. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap (Figure 6 and 7, Appendix B). The cassette should be positioned downward, perpendicular to the wind.
2. If AC or DC electricity is required then turn it on. If used, the generator should be placed 10 ft. downwind from the sampling pump.
3. Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.
4. Turn the pump on. Should intermittent sampling be required, sampling filters must be covered between active periods of sampling. To cover the sample filter: turn the cassette to face upward, place the cassette cap on the cassette, remove the inlet plug from the cassette cap, attach a rotameter to the inlet opening of the cassette cap to measure the flow rate, turn off the sampling pump, place the inlet plug into the inlet opening on the cassette cap. To resume sampling: remove the inlet plug, turn on the sampling pump, attach a rotameter to measure the flow rate, remove the cassette cap, replace the inlet plug in the cassette cap and invert the cassette, face downward and perpendicular to the wind.
5. Check the pump at sampling midpoint if sampling is longer than 4 hours. The generators may need to be regassed depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.



6. At the end of the sampling period, orient the cassette up, turn the pump off.
7. Check the flow rate as shown in Section 7.2.3. When sampling open-faced, the sampling cap should be replaced before post calibrating. Use the same cassette used for sampling for post calibration (increased dust/fiber loading may have altered the flow rate).
8. Record the post flow rate.
9. Record the cumulative time or run.
10. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

#### 7.4.3. Post Site Sampling

1. Follow handling procedures in Section 3.2, steps 1-4.
2. Obtain an electronic or hard copy of meteorological data which occurred during the sampling event. Record weather: wind speed, ambient temperature, wind direction, and precipitation. Obtaining weather data several days prior to the sampling event can also be useful.

### 7.5 Indoor Sampling Procedures

PCM analysis is used for indoor air samples. When analysis shows total fiber count above the OSHA action level 0.1 f/cc then TEM (U.S. EPA's Modified Yamate Method) is used to identify asbestos from non-asbestos fibers.

Sampling pumps should be placed four to five feet above ground level away from obstructions that may influence air flow. The pump can be placed on a table or counter. Refer to Table 2 (Appendix A) for a summary of indoor sampling locations and rationale for selection.

Indoor sampling utilizes high flow rates to increased sample volumes (2000 L for PCM and 2800 to 4200 L for TEM) in order to obtain lower detection limits below the standard, (i.e., 0.01 f/cc or lower [PCM])

and 0.005 structures/cc or lower [TEM]).

#### 7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail to detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. In the past, an 8-hour sampling period was recommended to cover various air circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The results from this sampling option typifies worst case condition. This is referred to as aggressive air sampling for asbestos. Refer to Table 2 for sample station locations.

1. Before starting the sampling pumps, direct forced air (such as a 1-horsepower leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should take at least 5 minutes per 1000 sq. ft. of floor.
2. Place a 20-inch fan in the center of the room. (Use one fan per 10,000 cubic feet of room space.) Place the fan on slow speed and point it toward the ceiling.
3. Follow procedures in Section 7.4.1 and 7.4.2 (Turn off the pump and then the fan(s) when sampling is complete.).
4. Follow handling procedures in Section 3.2, steps 1-4.

### 8.0 CALCULATIONS

The sample volume is calculated from the average flow rate of the pump multiplied by the number of minutes the pump was running (volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample (zero for lot, field and trip blanks).

The concentration result is calculated using the sample volume and the number of asbestos structures reported after the application of the cluster and matrix counting criteria.

## **9.0 QUALITY ASSURANCE/ QUALITY CONTROL**

Follow all QA/QC requirements from the laboratories as well as the analytical methods.

### **9.1 TEM Requirements**

1. Examine lot blanks to determine the background asbestos structure concentration.
2. Examine field blanks to determine whether there is contamination by extraneous asbestos structures during specimen preparation.
3. Examine of laboratory blanks to determine if contamination is being introduced during critical phases of the laboratory program.
4. To determine if the laboratory can satisfactorily analyze samples of known asbestos structure concentrations, reference filters shall be examined. Reference filters should be maintained as part of the laboratory's Quality Assurance program.
5. To minimize subjective effects, some specimens should be recounted by a different microscopist.
6. Asbestos laboratories shall be accredited by the National Voluntary Laboratory Accreditation Program.
7. At this time, performance evaluation samples for asbestos in air are not available for Removal Program Activities.

### **9.2 PCM Requirements**

1. Examine reference slides of known concentration to determine the analyst's ability to satisfactorily count fibers. Reference slides should be maintained as part of the laboratory's quality assurance program.
2. Examine field blanks to determine if there is contamination by extraneous structures during sample handling.

3. Some samples should be relabeled then submitted for counting by the same analyst to determine possible bias by the analyst.
4. Participation in a proficiency testing program such as the AIHA-NIOSH proficiency analytical testing (PAT) program.

## **10.0 DATA VALIDATION**

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives.

## **11.0 HEALTH AND SAFETY**

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures. More specifically, when entering an unknown situation involving asbestos, a powered air purifying respirator (PAPR) (full face-piece) is necessary in conjunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbestos concentrations are below personal health and safety levels, then Level D personal protection is adequate.

## **12.0 REFERENCES**

- (1) Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air, Part 1: Method, EPA/540/2-90/005a, May 1990, and Part 2: Technical Background Document, EPA/540/2-90/005b, May 1990.
- (2) Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, EPA's Report No. 68-02-3266, 1984, G. Yamate, S.C. Agarwal, and R. D. Gibbons.
- (3) National Institute for Occupational Safety and Health. NIOSH Manual of Analytical Method. Third Edition. 1987.
- (4) U.S. Environmental Protection Agency. Code of Federal Regulations 40 CFR 763. July 1, 1987. Code of Federal Regulations 40 CFR 763 Addendum. October 30, 1987.

(5) U.S. Environmental Protection Agency .  
Asbestos-Containing Materials in Schools ;  
Final Rule and Notice. 52 FR 41826.

(6) Occupational Safety and Health  
Administration. Code of Federal Regulations  
29 CFR 1910.1001. Washington, D.C .  
1987.

## APPENDIX A

### Tables

TABLE 1. SAMPLE STATIONS FOR OUTDOOR SAMPLING		
Sample Station Location	Sample Numbers	Rationale
Upwind/Background <sup>(1)</sup>	Collect a minimum of two simultaneous upwind/background samples 30 ° apart from the prevailing windlines.	Establishes background fiber levels.
Downwind	Deploy a minimum of 3 sampling stations in a 180 degree arc downwind from the source.	Indicates if asbestos is leaving the site.
Site Representative and/or Worst Case	Obtain one site representative sample which shows average condition on-site or obtain worst case sample (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

<sup>(1)</sup> More than one background station may be required if the asbestos originates from different sources.

## APPENDIX A (Cont'd)

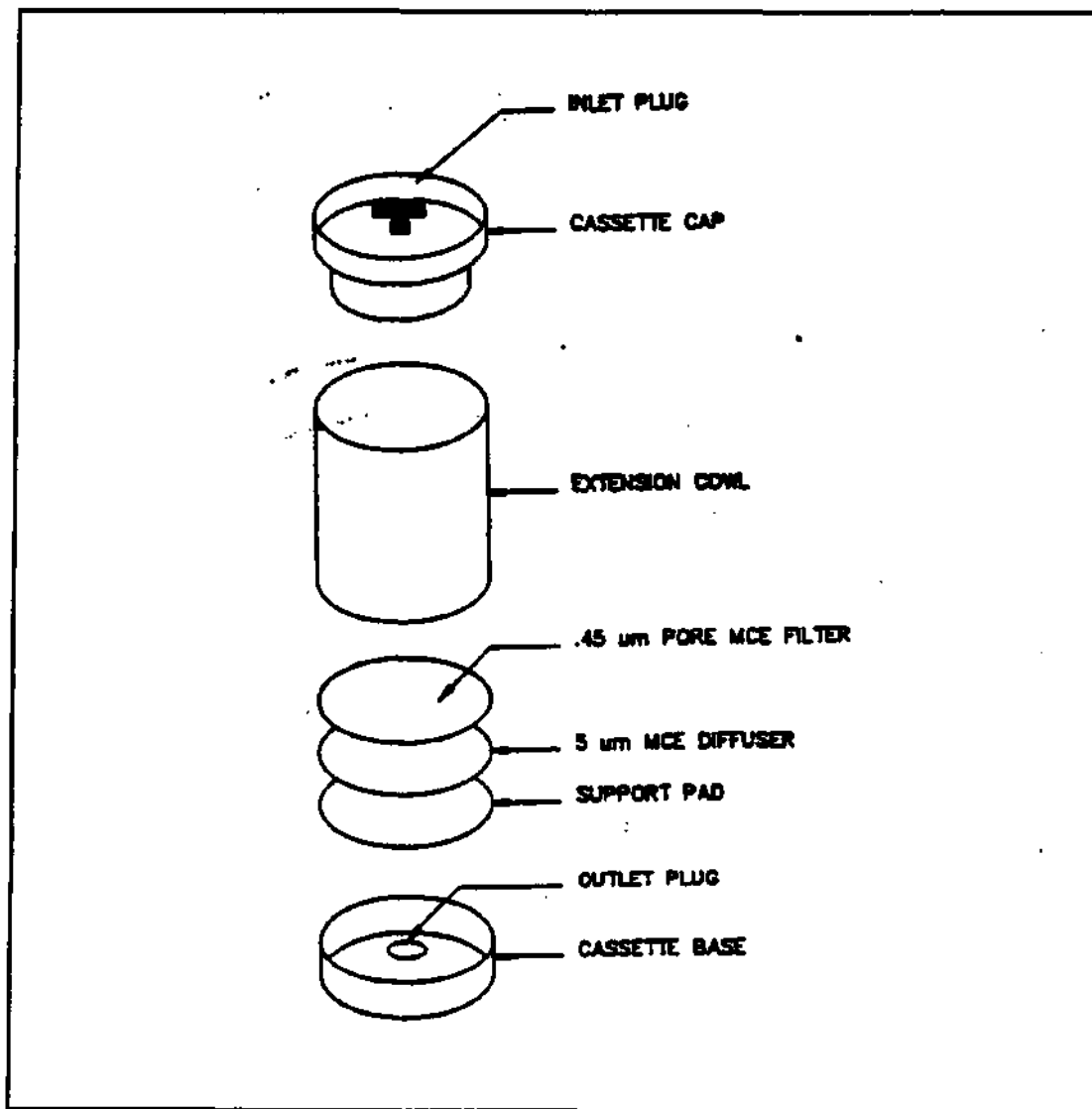
### Tables

TABLE 2 SAMPLE STATIONS FOR INDOOR SAMPLING		
Sample Station Location	Sample Numbers	Rationale
Indoor Sampling	<p>If a work site is a single room, disperse 5 samplers throughout the room.</p> <p>If the work site contains up to 5 rooms, place at least one sampler in each room.</p> <p>If the work site contains more than 5 rooms, select a representative sample of the rooms.</p>	Establishes representative samples from a homogeneous area.
Upwind/Background	If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30 ° apart from the prevailing windlines.	Establish whether indoor asbestos concentrations are coming from an outside source.
Worst Case	Obtain one worst case sample, i.e., aggressive sampling (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

## APPENDIX B

### Figures

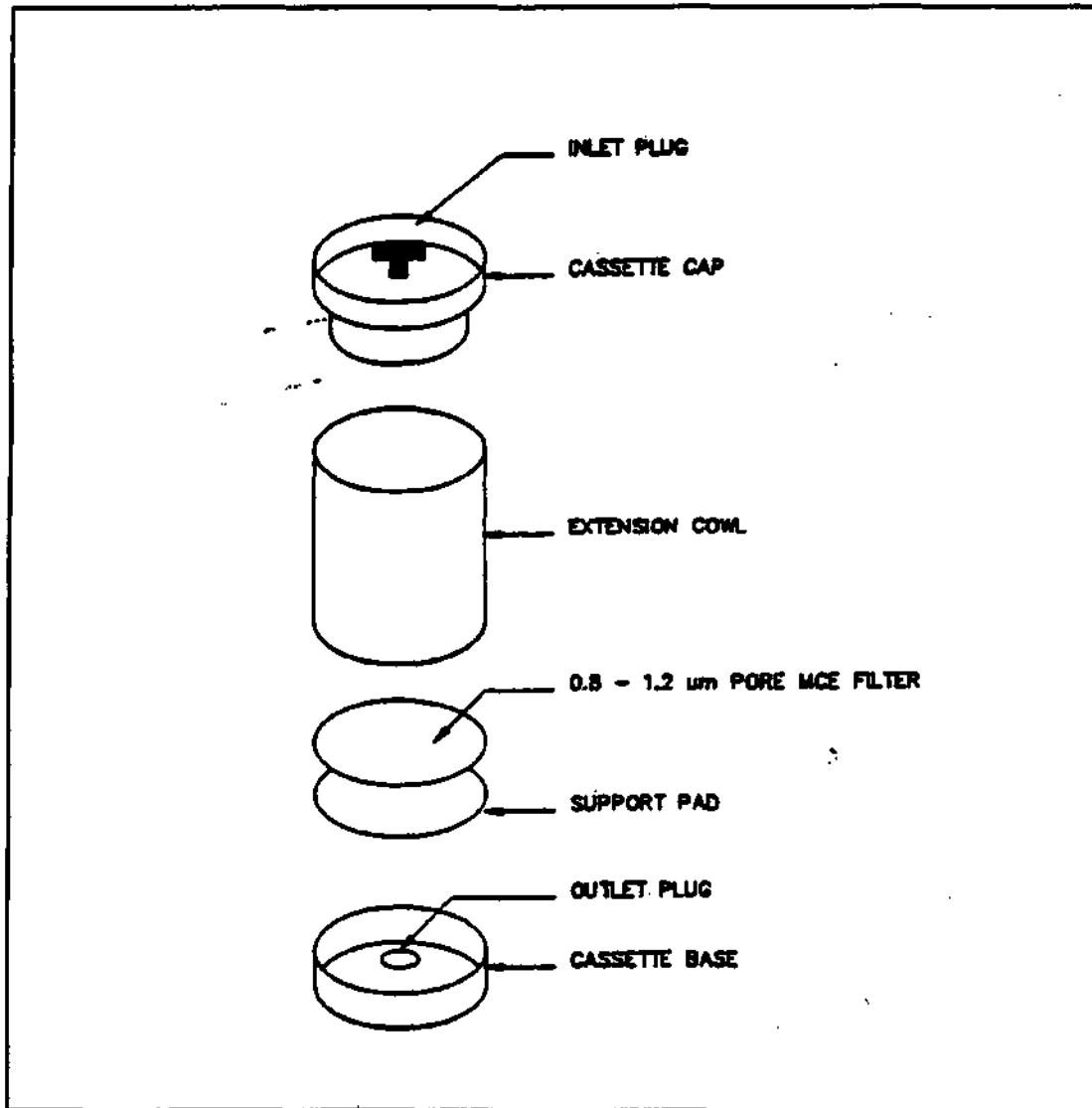
FIGURE 1. Transmission Electron Microscopy Filter Cassette



## APPENDIX B (Cont'd)

### Figures

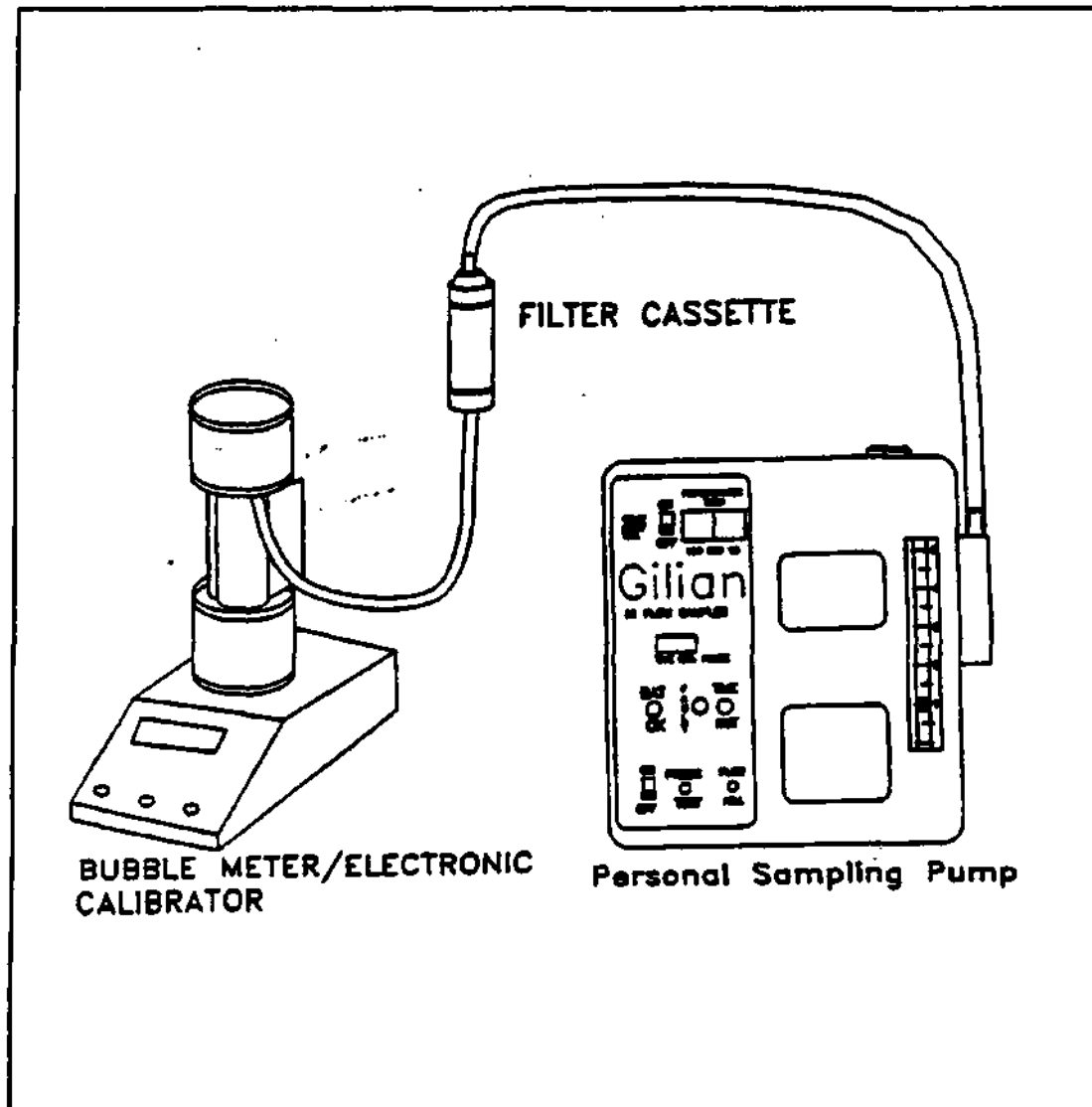
FIGURE 2. Phase Contrast Microscopy Filter Cassette



## APPENDIX B (Cont'd)

### Figures

FIGURE 3. Calibrating a Personal Sampling Pump with a Bubble Meter

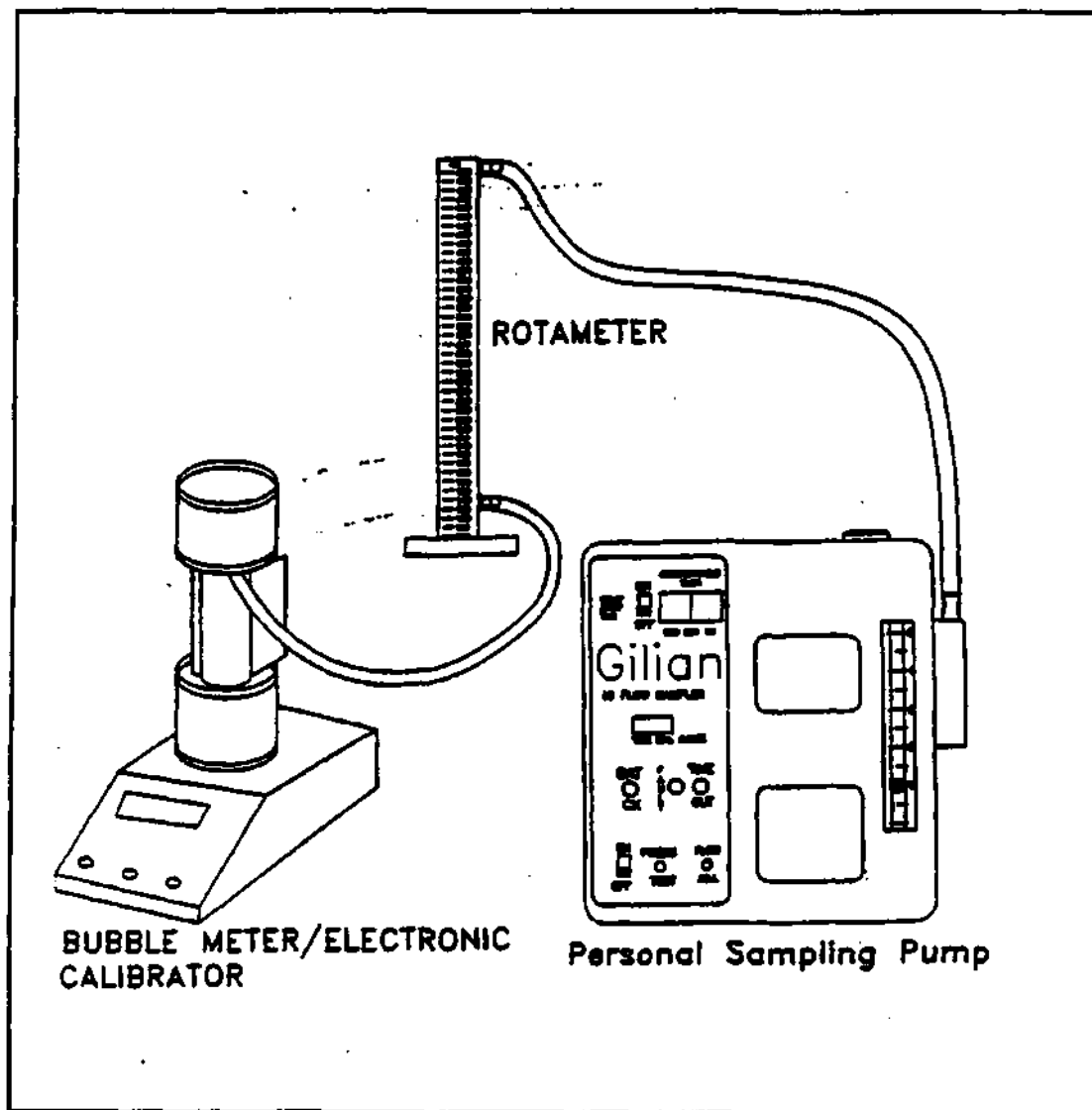




## APPENDIX B (Cont'd)

### Figures

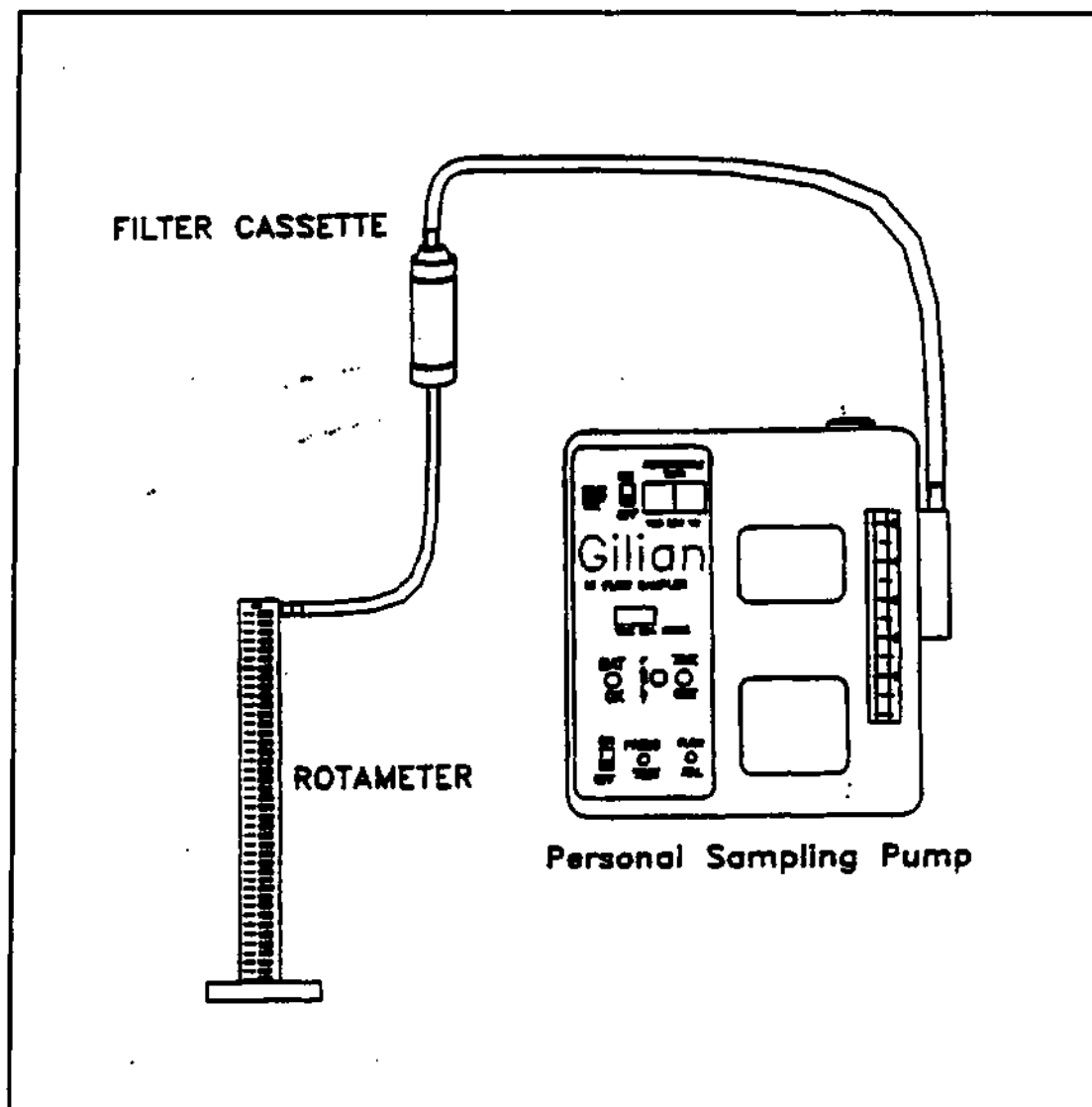
FIGURE 4. Calibrating a Rotameter with a Bubble Meter



## APPENDIX B (Cont'd)

### Figures

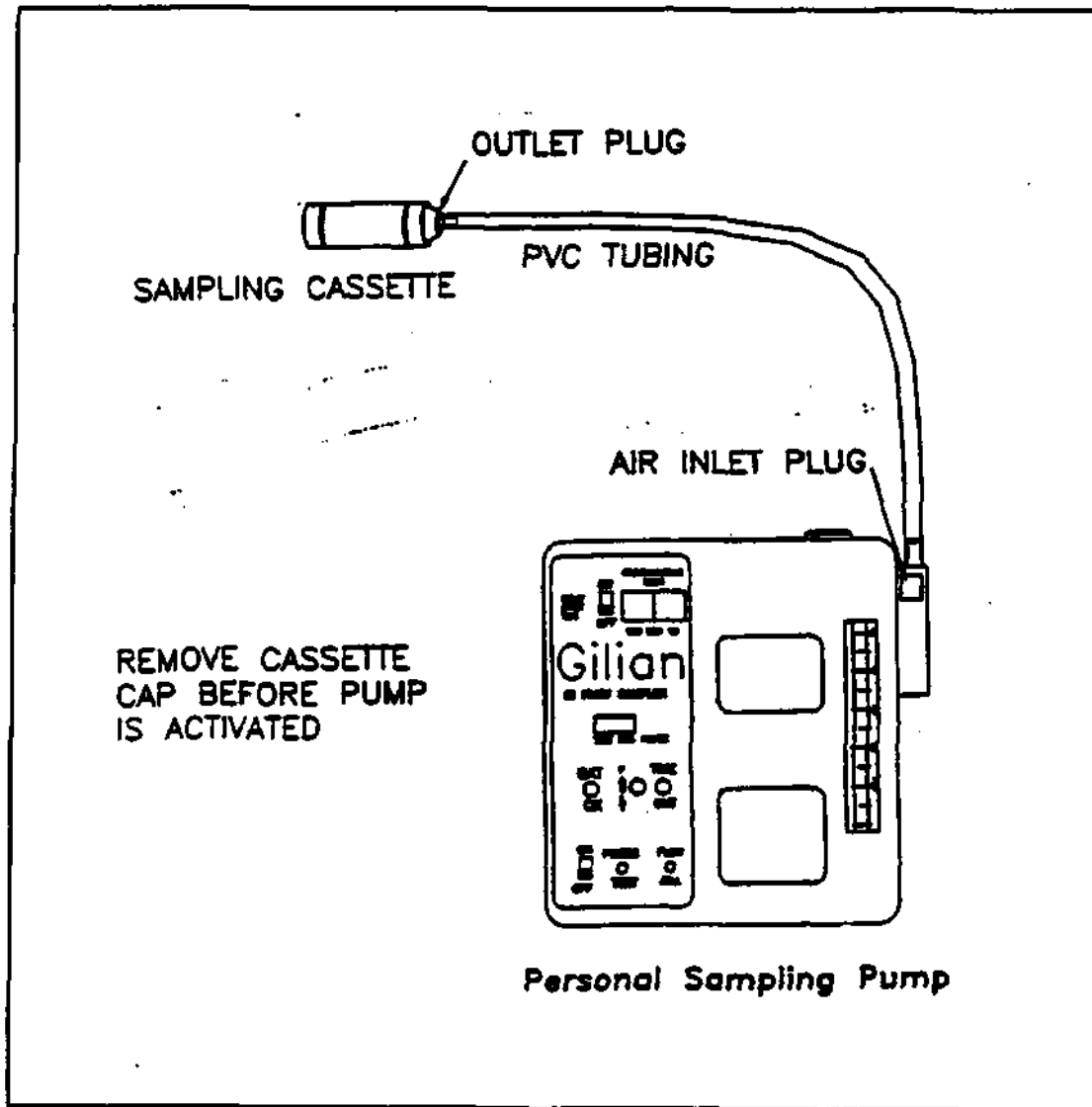
FIGURE 5. Calibrating a Sampling Pump with a Rotameter



## APPENDIX B (Cont'd)

### Figures

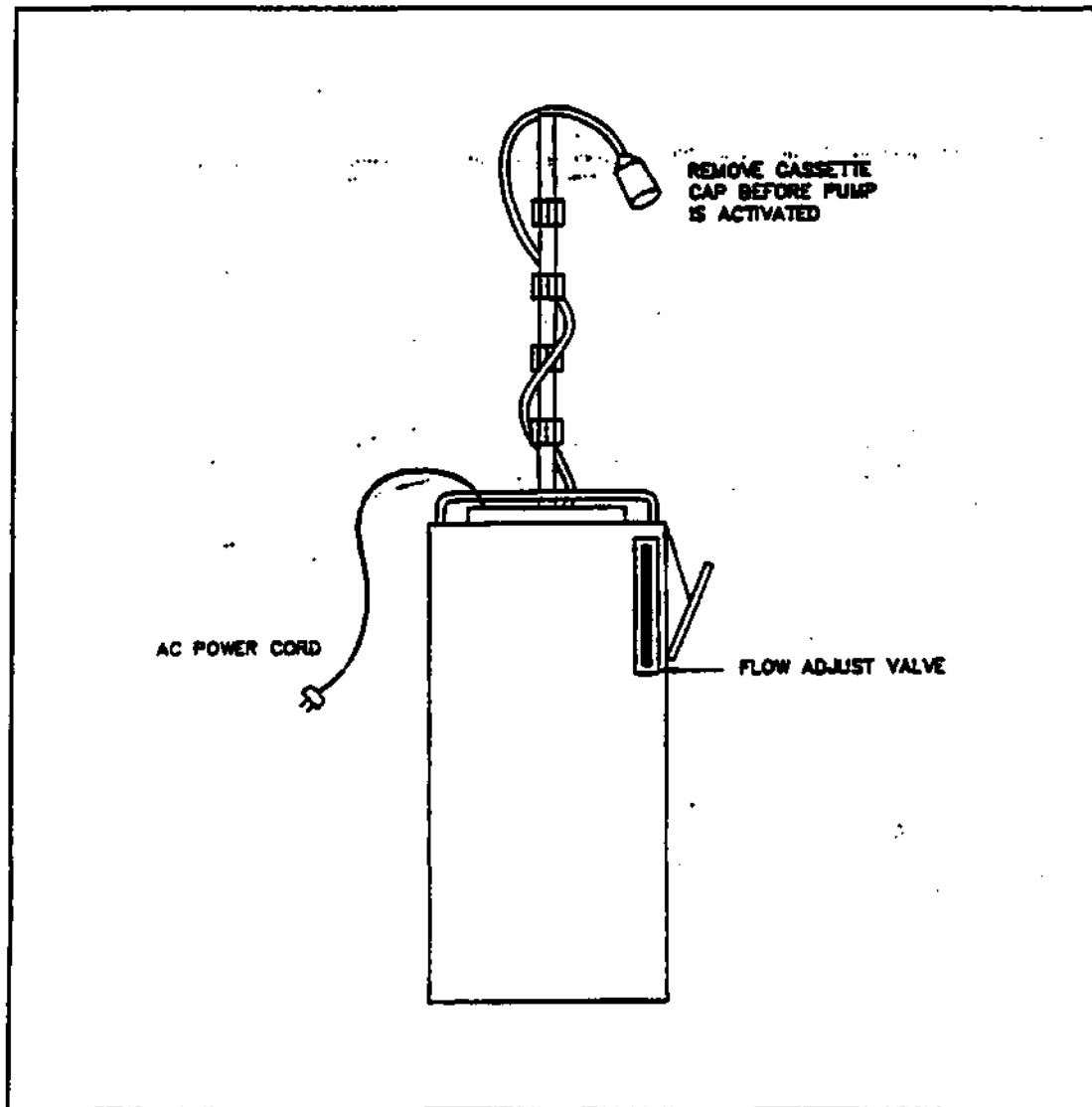
FIGURE 6. Personal Sampling Train for Asbestos



## APPENDIX B (Cont'd)

### Figures

FIGURE 7. High Flow Sampling Train for Asbestos





## Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations<sup>1</sup>

This standard is issued under the fixed designation D 5755; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers a procedure to (a) identify asbestos in dust and (b) provide an estimate of the concentration of asbestos in the sampled dust reported as the number of asbestos structures per unit area of sampled surface.

1.1.1 If an estimate of the asbestos mass is to be determined, the user is referred to Test Method D 5756.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos structures. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, electrostatic properties and other factors.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the concentration of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices that can be more accurately quantified by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the

responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 1193 Specification for Reagent Water<sup>2</sup>
- D 1739 Test Method for the Collection and Measurement of Dustfall (Settleable Particulate Matter)<sup>3</sup>
- D 3193 Practice for Rotameter Calibration<sup>3</sup>
- D 3670 Guide for Determination of Precision and Bias of Methods of Committee D-22<sup>3</sup>
- D 5756 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration<sup>3</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs (1),<sup>4</sup> (2) and (3).

3.1.2 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate dominated minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

**Discussion**—Included in the definition are the asbestiform varieties of: serpentine (chrysotile); riebeckite (crocidolite); grunerite (grunerite asbestos); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association (3).

Asbestos	Chemical Abstract Service No. <sup>5</sup>
Chrysotile	12001-29-3
Crocidolite	12001-28-4
Grunerite Asbestos	12172-73-3
Anthophyllite Asbestos	77336-47-3
Tremolite Asbestos	77336-48-6
Actinolite Asbestos	77336-46-4

3.1.3 *fibril*—a single fiber that cannot be separated into

<sup>1</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>2</sup> Annual Book of ASTM Standards, Vol 11.03.

<sup>3</sup> The boldface numbers in parentheses refer to the list of references at the end of this test method.

<sup>4</sup> The non-asbestiform variations of the minerals indicated in 3.1.3 have different Chemical Abstract Service (CAS) numbers.

<sup>5</sup> This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

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smaller components without losing its fibrous properties or appearance.

### 3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

3.2.2 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.3 *cluster*—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.

3.2.5 *dust*—any material composed of particles in a size range of  $\leq 1$  mm and large enough to settle by virtue of their weight from the ambient air (see definition for settleable particulate matter in Test Method D 1739).

3.2.6 *fiber*—a structure having a minimum length of 0.5  $\mu\text{m}$ , an aspect ratio of 5:1 or greater, and substantially parallel sides (4).

3.2.7 *fibrous*—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by a single particle or connected group of non-fibrous particles. The exposed fiber must meet the fiber definition (see 3.2.6).

3.2.10 *structures*—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices). Final results of the test are always expressed in asbestos structures per square centimetre.

## 4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous solution of known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification of 15 000 to 20 000X.

## 5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an index of the concentration of asbestos structures in the dust per unit area analyzed as derived from a quantitative TEM analysis.

5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

5.1.2 At present, a single direct relationship between asbestos-containing dust and potential human exposure does not exist. Accordingly, the user should consider these data in relationship to other available information in their evaluation.

5.2 This test method uses the definition, settleable particulate material, found in Test Method D 1739 as the definition of dust. This definition accepts all particles small enough to pass through a 1 mm (No. 18) screen. Thus, a single, large asbestos containing particle(s) (from the large end of the particle size distribution) dispersed during sample preparation may result in anomalously large asbestos concentration results in the TEM analyses of that sample. It is, therefore, recommended that multiple independent samples are secured from the same area, and a minimum of three samples analyzed by the entire procedure.

## 6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite.*

6.1.2 *Palygorskite (Attapulgite).*

6.1.3 *Halloysite.*

6.1.4 *Pyroxenes.*

6.1.5 *Sepiolite.*

6.1.6 *Vermiculite scrolls.*

6.1.7 *Fibrous talc.*

6.1.8 Hornblende and other amphiboles other than those listed in 3.1.2.

6.2 Collecting any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, must be avoided.

## 7. Materials and Equipment

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without

lessening the accuracy of the determination.<sup>6</sup>

7.2 *Transmission Electron Microscope (TEM)*, an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

7.3 *Energy Dispersive X-ray System (EDXA)*.

7.4 *High Vacuum Carbon Evaporator*, with rotating stage.

7.5 *High Efficiency Particulate Air (HEPA)*, filtered negative flow hood.

7.6 *Exhaust or Fume Hood*.

7.7 *Particle-free Water* (ASTM Type II, see Specification D 1193).

7.8 *Glass Beakers* (50 mL).

7.9 *Glass Sample Containers*, with wide mouth screw cap (200 mL) or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.10 *Waterproof Markers*.

7.11 *Forceps* (tweezers).

7.12 *Ultrasonic Bath*, table top model (100 W).

7.13 *Graduated Pipettes* (1, 5, 10 mL sizes), glass or plastic.

7.14 *Filter Funnel*, either 25 mm or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.15 *Side Arm Filter Flask*, 1000 mL.

7.16 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47 mm diameter,  $\leq 0.22 \mu\text{m}$  and  $5 \mu\text{m}$  pore size.

7.17 *Polycarbonate (PC) Filters*, 25 or 47 mm diameter,  $\leq 0.2 \mu\text{m}$  pore size.

7.18 *Storage Containers*, for the 25 or 47 mm filters (for archiving).

7.19 *Glass Slides*, approximately 76 by 25 mm in size.

7.20 *Scalpel Blades*, No. 10, or equivalent.

7.21 *Cabinet-type Desiccator*, or low temperature drying oven.

7.22 *Chloroform*, reagent grade.

7.23 *Acetone*, reagent grade.

7.24 *Dimethylformamide (DMF)*.

7.25 *Glacial Acetic Acid*.

7.26 *1-methyl-2-pyrrolidone*.

7.27 *Plasma Asher*, low temperature.

7.28 *pH Paper*.

7.29 *Air Sampling Pump*, low volume personal-type, capable of achieving a flow rate of 1 to 5 L/min.

7.30 *Rotameter*.

7.31 *Air Sampling Cassettes*, 25 mm or 37 mm, containing  $0.8 \mu\text{m}$  or smaller pore size MCE or PC filters.

7.32 *Cork Borer*, 7 mm.

7.33 *Non-Asbestos Mineral*, references as outlined in 6.1.

7.34 *Asbestos Standards*, as outlined in 3.1.2.

7.35 *Tygon<sup>7</sup> Tubing*, or equivalent.

7.36 *Small Vacuum Pump*, that can maintain a pressure of 92 kPa.

7.37 *Petri Dishes*, large glass, approximately 90 mm in diameter.

7.38 *Jaffe Washer*, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 mm by 50 mm in size.

7.39 *Copper TEM Finder Grids*, 200 mesh.

7.40 *Carbon Evaporator Rods*.

7.41 *Lens Tissue*.

7.42 *Ashless Filter Paper Filters*, 90 mm diameter.

7.43 *Gummed Paper Reinforcement Rings*.

7.44 *Wash Bottles*, plastic.

7.45 *Reagent Alcohol*, HPLC Grade (Fisher A995 or equivalent).

7.46 *Opening Mesh Screen*, plastic, 1.0 by 1.0 mm, (Spectra-Mesh #146410 or equivalent).

7.47 *Diffraction Grating Replica*.

## 8. Sampling Procedure for Microvacuum Technique

8.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to  $0.8 \mu\text{m}$  are required (7.31). The number of samples collected depends upon the specific circumstances of the study.

8.2 Maintain a log of all pertinent sampling information and sampling locations.

8.3 Sampling pumps and flow indicators shall be calibrated using a certified standard apparatus or assembly (see Practice D 3195 and 7.29).

8.4 Record all calibration information (5).

8.5 Perform a leak check of the sampling system at each sampling site by activating the pump (7.29) with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

8.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (7.35). The plastic tubing must be long enough in that the sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity is 100 ( $\pm 10$ ) cm/s. This air velocity calculation is based on an internal sampling tube diameter of 6.35 mm at a flow rate of 2 L/min.

8.7 Measure and determine the sample area of interest. A

<sup>6</sup> *Reagent Chemicals*, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

<sup>7</sup> Tygon is a registered trademark of the DuPont Co.

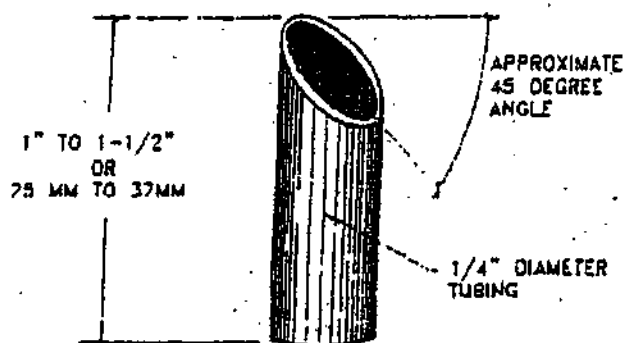


FIG. 1 Example of the Tubing Nozzle

sample area of 100 cm<sup>2</sup> is vacuumed until there is no visible dust or particulates matter remaining. Perform a minimum of two orthogonal passes on the surface within a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 4.2).) Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm<sup>2</sup>. Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

8.8 At the end of sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end-plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved and rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

8.10 Wipe off the exterior surface of the cassettes with disposable wet towels (baby wipes) prior to packaging for shipment.

## 9. Sample Shipment

9.1 Ship dust samples to an analytical laboratory in a sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic "bubble pack" is probably the most appropriate material for this purpose.

## 10. Sample Preparation

10.1 Under a negative flow HEPA hood (7.5), carefully wet-wipe the exterior of the cassettes to remove any possible

contamination before taking cassettes into a clean preparation area.

10.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

10.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank concentrations (see Section 11).

10.4 All sample preparation steps 10.4.1 through 10.4.6 shall take place in the dust preparation area inside a HEPA hood.

10.4.1 Remove the upper plug from the sample cassette and carefully introduce approximately 10 mL solution of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle (7.44). If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol solution into the cassette through the tubing, and then remove the tubing, if it is visibly clean.

10.4.2 Replace the upper plug or the sample cap and lightly shake the dust suspension by hand for 3 s.

10.4.3 Remove the entire cap of the cassette and pour the suspension through a 1.0 by 1.0 mm opening screen (7.46) into a pre-cleaned 200 mL glass specimen bottle (7.9). All visible traces of the sample contained in the cassette shall be rinsed through the screen into the specimen bottle with a plastic wash bottle containing the 50/50 solution of particle-free water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Discard the 1.0 by 1.0 mm screen and bring the volume of solution in the specimen bottle up to the 100 mL mark on the side of the bottle with particle-free water only.

10.4.4 Adjust the pH of the suspension to 3 to 4 using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

10.4.5 Use either a disposable plastic filtration unit or a glass filtering unit (7.14) for filtration of aliquots of the suspension. The ability of an individual filtration unit to produce a uniform distribution may be tested by the filtration of a colored particulate suspension such as diluted India ink (suspension of carbon black).

10.4.5.1 If a disposable plastic filtration unit is used, then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side-arm vacuum flask (7.15). Place a 5.0 µm pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE (7.16) or PC filter (≤0.22 µm pore size) (7.17) on top of the backing filter. Apply a vacuum (7.36), ensuring



that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires the discard of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

10.4.5.2 If a glass filtration unit is used, place a 5  $\mu\text{m}$  pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter ( $\leq 0.22 \mu\text{m}$  pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample aliquots, prepare a blank filter by filtration of 50 mL of particle-free water. If aliquots of the same sample are filtered in order of increasing concentration, the glass filtration unit need not be washed between filtration. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual suspension from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in particle-free water.

10.4.6 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

10.4.7 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath (7.12) and sonicate for 3.0 min. Maintain the water level in the sonicator at the same height as the solution in sample bottle. The ultrasonic bath shall be calibrated as described in 20.5. The ultrasonic bath must be operated at equilibrium temperature. After sonicating, return the sample bottle to the work surface of the HEPA hood. Preparation steps 10.4.8 through 10.4.14 shall be carried out in this hood.

10.4.8 Shake the suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

10.4.9 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. If after examination in the TEM, the smallest volume measured (1.0 mL) (7.13) yields an overloaded sample, then perform additional serial dilutions of the suspension. If it is estimated that less than 1.0 mL of solution has to be filtered because of the density of the suspension, perform a serial dilution.

10.4.9.1 If serial dilutions are required, repeat step 10.4.8 before the serial dilution portion is taken. Do not re-sonicate the original solution or any serial dilutions. The recommended procedure for a serial dilution is to mix 10 mL of the sample solution with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing dilutions.

10.4.10 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the water in the funnel.

10.4.11 Apply vacuum to the flask and draw the mixture through the filter.

10.4.12 Discard the pipette.

10.4.13 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers (7.11). Place the completed sample filter particle side up, into a precleaned, labeled, disposable, plastic petri dish (7.48) or other similar container.

10.4.14 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different aliquots of the dust suspension. For this series of filters, it is recommended that the volume of each aliquot of the original suspension be a factor of five higher than the previous one. If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

10.4.15 There are many practical methods for drying MCE filters. The following are two examples that can be used: (1) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator (7.21); (2) to shorten the drying time (if desired), remove a plug of the damp filter and attach it to a glass slide (7.19) as described in 12.1.2 and 12.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

10.4.16 PC filters do not require lengthy drying before preparation, but shall be placed in a desiccator for at least 30 min before preparation.

10.5 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

## 11. Blanks

11.1 Prepare sample blanks that include both a process blank (50 mL of particle-free water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC) used in the laboratory. If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. Blanks will be considered contaminated, if after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

11.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

## 12. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

NOTE 1—Use of either the acetone or the dimethylformamide-acetic acid method is acceptable.

### 12.1 Acetone Fixing Method:

12.1.1 Remove a section (a plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer (7.32). The cork borer must be wet wiped after each time a section is removed.

12.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement (7.43), or other suitable means. Label the slide with a glass scribing tool or permanent marker (7.10).

12.1.3 Prepare a fusing dish from a glass petri dish (7.37) and a metal screen bridge (7.38) with a pad of five to six ashless paper filters (7.42) and place in the bottom of the petri dish (4). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

#### 12.2 Dimethylformamide-Acetic Acid Method:

12.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the clearing solution just saturates the filter section.

12.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

12.3 Plasma etching of the collapsed filter is required.

12.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher (7.27). Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm<sup>3</sup>/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the *USEPA Asbestos-Containing Materials in Schools* (4) or *NIST/NVLP Program Handbook for Airborne Asbestos Analysis* (6) documents.

12.3.2 Place the glass slide containing the collapsed filters into the low-temperature plasma asher, and etch the filter.

12.4 Carbon coating of the collapsed and etched filters is required.

12.4.1 Carbon coating must be performed with a high-vacuum coating unit (7.4), capable of less than 10<sup>-4</sup> torr (13 MPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall

not be used. Carbon rods (7.40) used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 3 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

12.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 MPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen.

12.4.2.1 If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

12.5 Preparation of the Jaffe Washer—The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used (7, 8). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

12.5.1 Place several pieces of lens tissue (7.41) on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 2.

#### 12.6 Placing the Specimens into the Jaffe Washer:

12.6.1 Place the TEM grids (7.39) shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

12.6.2 Prepare three grids from each sample.

12.6.2.1 Using a curved scalpel blade (7.20), excise at least two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

12.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

12.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

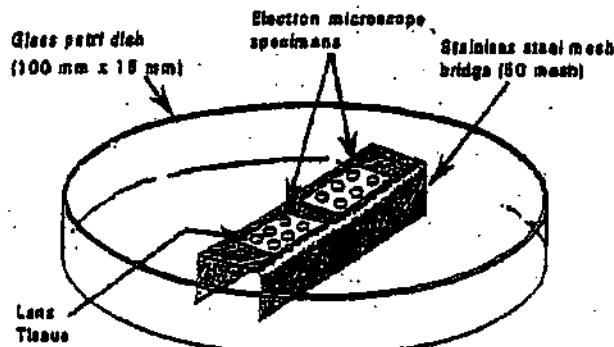


FIG. 2 Example of Design of Solvent Washer (Jaffe Washer)

12.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

12.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

12.7 Alternately, place the grids on a low level (petri dish filled to the 1/4 mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

12.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

### 13. TEM Specimen Preparation of Polycarbonate (PC) Filter

13.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

13.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

13.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label each PC strip with the individual sample number.

13.4 Carbon coat the PC filter strips as discussed in 12.4.2. PC filters do not require etching.

**NOTE 2: Caution**—Do not overheat the filter sections while carbon coating.

13.5 Prepare a Jaffe washer as described in 12.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

13.6 Using a clean curved scalpel blade, excise three, 3-mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

13.7 Place the lid on the Jaffe washer and rest the grids in place for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, marked grid box.

### 14. Grid Opening Measurements

14.1 TEM grids must have a known grid opening area. Determine this area as follows:

14.2 Measure at least 20 grid openings in each of 20 random 75 to 100  $\mu\text{m}$  (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

14.3 Grid area measurements can also be made at the

TEM at a calibrated screen magnification of between 15 000 and 20 000X. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

14.4 Pre-calibrated TEM grids are also acceptable for this test method.

### 15. TEM Method

15.1 Microscope settings: 80 to 120 kV, 15 000 to 20 000X screen magnification for analysis (7.2).

15.2 Analyze two grids for each sample. Analyze one-half of the sample area on one sample grid preparation and the remaining half on a second sample grid preparation.

#### 15.3 Determination of Specimen Suitability:

15.3.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the X and y translation directions of the microscope. Insert the specimen holder into the microscope.

15.3.2 Scan the entire grid at low magnification (250X to 1000X) to determine its suitability for high magnification analysis as specified in 15.3.3.

15.3.3 Grids are acceptable for analysis if the following conditions are met:

15.3.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

15.3.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

15.3.3.3 The fractional area of undissolved filter is less than 10 %.

15.3.3.4 The fraction of grid openings with overlapping or folded replica film is less than 50 %.

15.3.3.5 At least 20 grid openings, that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

#### 15.4 Determination of Grid Opening Suitability:

15.4.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening prior to the analysis.

15.4.2 The individual grid opening must have less than 5 % holes over its area.

15.4.3 Grid openings must be less than 25 % covered with particulate matter.

15.4.4 Grid openings must be uniformly loaded.

15.5 Observe and record the orientation of the grid at 80 to 150X, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

### 16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the *USEPA Asbestos-Containing*

*Materials in Schools* document (4). Record, for each AHERA structure identified, the length and width measurements.

16.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

16.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis (7.3) and recorded on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

16.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al. (7) or Chatfield and Dillon (8).

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

#### 16.7 Counting Rules

16.7.1 At a screen magnification of between 15 000 and 20 000X evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

16.8 *Analytical Sensitivity*—An analytical sensitivity of approximately 1000 asbestos structures per square centimetre (calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, or decreasing the size of the final filter. Occasionally, due to high particle loadings or high asbestos concentration, this analytical sensitivity cannot be practically achieved and stopping rules apply.

16.9 *Limit of Detection*—The limit of detection for this method is defined as, at a minimum, the counting of four asbestos structures during the TEM analysis. If less than four asbestos structures are counted during the analysis then the analytical result which will be reported will be less than the limit of detection and a "less than" sign (<) will appear before the number. All data shall be provided in the laboratory report.

#### 16.10 Stopping Rules

16.10.1 The analysis is stopped upon the completion of the grid square that achieves an analytical sensitivity of less than 1000 asbestos structures per square centimetre.

16.10.2 If an analytical sensitivity of 1000 asbestos structures per square centimetre cannot be achieved after analyzing ten grid openings then stop on grid opening No. 10 or the grid opening which contains the 100th asbestos structure, whichever comes first. A minimum of four grid squares shall be analyzed for each sample.

16.10.2.1 If the analysis is stopped because of the 100th structure rule, the entire grid square containing the 100th structure must be counted.

16.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

## 17. Sample Storage

17.1 The washed-out sample cassettes can be discarded after use.

17.2 Sample grids and unused filter sections (7.18) must be stored for a minimum of one year.

## 18. Reporting

18.1 Report the following information for each dust sample analyzed:

18.1.1 Concentration in structures/cm<sup>2</sup>.

18.1.2 The analytical sensitivity.

18.1.3 Types of asbestos present.

18.1.4 Number of asbestos structures counted.

18.1.5 Effective filtration area.

18.1.6 Average size of the TEM grid openings that were counted.

18.1.7 Number of grid openings examined.

18.1.8 Sample dilution used.

18.1.9 Area of the surface sampled.

18.1.10 Listing of size data for each structure counted.

18.1.11 A copy of the TEM count sheet or a complete listing of the raw data. An example of a typical count sheet is shown in Appendix X1.

18.2 Determine the amount of asbestos in any accepted sample using the following formula:

$$\frac{EFA \times 100 \text{ mL} \times \#STR}{GO \times GOA \times V \times SPL} = \text{asbestos structures/cm}^2 \quad (1)$$

where:

#STR = number of asbestos structures counted;

EFA = effective filter area of the final sampling filter, mm<sup>2</sup>;

GO = number of grid openings counted;

GOA = average grid opening area, mm<sup>2</sup>;

SPL = surface area sampled, cm<sup>2</sup>; and

V = volume of sample filtered, in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL.

## 19. Quality Control/Quality Assurance

19.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

19.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The re-analysis shall consist of a second sample preparation obtained from the final filter.

19.2 In addition, quality assurance programs must follow the criteria shown in the *USEPA Asbestos-Containing Materials in Schools* document (4) and in the *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* document (6). These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

## 20. Calibrations

20.1 Perform calibrations of the instrumentation on a

regular basis, and retain these records in the laboratory, in accordance with the laboratory's quality assurance program.

20.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

20.3 A calibration list for the instrument is as follows:

20.3.1 TEM:

20.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

20.3.1.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

20.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (7.47) (for example, one containing at least 2160 lines/mm).

(a) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(b) Frequency of calibration will depend on the service history of the particular microscope.

(c) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(d) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(e) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

20.3.1.4 The smallest spot size of the TEM must be checked.

(a) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000X. An exposure time of 1 s is usually adequate.

(b) The measured spot size must be less than or equal to 250 nm.

20.4 EDXA:

20.4.1 The resolution and calibration of the EDXA must be verified.

20.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

20.4.1.2 Compare the X-ray energy versus channel

number for the Cu peak and be certain that readings are within  $\pm 10$  eV.

20.4.2 Collect a standard EDXA of crocidolite asbestos (NIST SRM 1866).

20.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

20.4.3 Collect a standard EDXA of chrysotile asbestos.

20.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

20.5 Ultrasonic bath calibration shall be performed as follows:

20.5.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for the dust analysis. Operate the bath until the water reaches the equilibrium temperature.

20.5.2 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

20.5.4 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature.

20.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{(\theta_2 - \theta_1)}{t} \quad (2)$$

where:

4.185 = Joules/cal,

R = energy deposition, watts/mL,

$\theta_1$  = temperature rise with the ultrasonic bath not operating, °C,

$\theta_2$  = temperature rise with the ultrasonic bath operating, °C,

t = time in seconds, 60 s (20.5.3 and 20.5.5),

$\sigma$  = specific heat of the liquid in the glass sample container, 1.0 cal/g, and

$\rho$  = density of the liquid in the glass sample container, 1.0 g/cm<sup>3</sup>.

20.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m<sup>2</sup>, as defined by this procedure.

## 21. Precision and Bias

21.1 *Precision*—The precision of the procedure in this test method is being determined using round robin data from participating laboratories.

21.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined (see Specification D 3670).

NOTE 3—Round robin data is under development and will be presented as a research report.

## 22. Keywords

22.1 asbestos; microvacuuming; settled dust; TEM

APPENDIX

(Nonmandatory Information)

X1. DUST SAMPLE ANALYSIS

X1.1 See Figs. X1.1 and X1.2 for the dust analysis worksheet and the TEM count sheet.

DUST SAMPLE ANALYSIS

Client: \_\_\_\_\_  
 Sample ID: \_\_\_\_\_  
 Job Number: \_\_\_\_\_  
 Date Sample Analyzed: \_\_\_\_\_  
 Number of Openings/Grids Counted: \_\_\_\_\_  
 Grid Accepted, 600X: Yes No  
 Percent Loading: \_\_\_\_\_ %  
 Grid Box #1: \_\_\_\_\_

Accelerating Voltage: \_\_\_\_\_  
 Indicated Mag: \_\_\_\_\_ KX  
 Screen Mag: \_\_\_\_\_ KX  
 Microscope: 1 2 3 4 5  
 Filter Type: \_\_\_\_\_  
 Filter Size: \_\_\_\_\_  
 Filter Pore Size (μm): \_\_\_\_\_  
 Grid Opening: 1) \_\_\_\_\_ μm x \_\_\_\_\_ μm  
 2) \_\_\_\_\_ μm x \_\_\_\_\_ μm

Analyst: \_\_\_\_\_

Reviewer: \_\_\_\_\_

Counting Rules: AHERA LEVEL II

Calculation Data:

Effective Filter Area in mm<sup>2</sup>: (EFA) \_\_\_\_\_  
 Number of Grid Openings Counted: (GO) \_\_\_\_\_  
 Average Grid Opening Area in mm<sup>2</sup>: (GOA) \_\_\_\_\_  
 Volume of sample Filtered in ml: (V) \_\_\_\_\_  
 Surface area Sampled in cm<sup>2</sup>: (SPL) \_\_\_\_\_  
 Number of Asbestos Structures Counted: (#STR) \_\_\_\_\_

\* If the number of asbestos structures counted is less than or equal to 4, enter 4 structures as the limit of detection here.

FORMULA FOR CALCULATION OF ASBESTOS STRUCTURES "DUST" PER CM<sup>2</sup>:

$$\frac{EFA \times 100 \times \#STR}{GO \times GOA \times V \times SPL} = (\text{Asbestos Structures per cm}^2)$$

Results for Total Asbestos Structures: \_\_\_\_\_  
 (Structures per cm<sup>2</sup>)

Results for Structures ≥ microns: \_\_\_\_\_  
 (Structures per cm<sup>2</sup>)

Job Number: \_\_\_\_\_

[illegible]

**Note: Keys to Abbreviations Used in Figure:**

Type:	
C	Chrysotile
AM	Amosite
CR	Crocidolite
AC	Actinolite
TR	Tremolite
AN	Anthrophyllite
N	Non Asbestos

**Structure:**  
**F** = Fiber  
**B** = Bundle  
**C** = Cluster  
**M** = Matrix

**Others:**

NSD	=	No Structures Detected
Morph	=	Morphology
SAED	=	Selected Area Electron Diffraction
EDS	=	Energy Dispersive X-Ray Spectroscopy
ER	=	Inter-Row Spacing
NP	=	No Pattern

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- (5) OSHA, *OSHA Technical Manual, OSHA Instruction CPL 2-208*, Directorate of Technical Support, U.S. Department of Labor, Washington, DC 20210, Feb. J. 1990, pp. 1-8 to 1-11.
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**Appendix C**

**Site Health and Safety Plan**

**HEALTH AND SAFETY PLAN FORM**

CDM FEDERAL PROGRAMS CORPORATION

CDM FPC Health and Safety Program

PROJECT DOCUMENT #: 2603-23-HSAP

**PROJECT NAME:** Libby Sister Sites (Asbestos Project) - Emergency Response**WORK ASSIGNMENT #:** Task Order No. C0023**REGION:** VIII**JOBSITE ADDRESS:** 733 West 800 South and 333 West 100 South**CLIENT:** USDOT Volpe National Transportation Systems CenterSalt Lake City, UT**PROJECT:** Former Vermiculite Intermountain Sites (SLC1 and SLC2)**DOT CONTACT:** John McGuiggin (TOCOR)**EPA CONTACT:** Joyce Ackerman and Floyd Nichols, EPA OSC**PHONE#:** (617) 494-2574**PHONE#:** (303) 312-6822 and (303) 312-6983, respectively**OBJECTIVES OF FIELD WORK:**

The purpose of this sampling effort is to acquire information useful for the design of more comprehensive emergency response investigations to assess the magnitude and extent of exposure to toxicologically relevant asbestos fibers at the Libby Sister Sites.

**TYPE:** Check as many as applicable

- |   |  |   |
|---|--|---|
| <input type="checkbox"/> Active               | <input type="checkbox"/> Landfill                | <input type="checkbox"/> Unknown            |
| <input checked="" type="checkbox"/> Inactive  | <input checked="" type="checkbox"/> Uncontrolled | <input type="checkbox"/> Military           |
| <input type="checkbox"/> Secure               | <input checked="" type="checkbox"/> Industrial   | <input type="checkbox"/> Other: Residential |
| <input checked="" type="checkbox"/> Unsecured | <input type="checkbox"/> Recovery                |   |
| <input type="checkbox"/> Enclosed Space       | <input type="checkbox"/> Well Field              |   |

**DESCRIPTION AND FEATURES:**

According to historical mining records, 80 percent of the world's vermiculite came from the Zonolite Mountains in Libby, Montana. EPA has determined that the vermiculite ore that was mined from these mountains is contaminated with amphibole asbestos. This ore was shipped throughout the United States both as processed and unprocessed material to locations including Salt Lake City, UT (Figure 1A and 1B). EPA has been conducting various investigations to determine potentially contaminated properties, outside Libby, which may have resulted from the Libby mining operations. This asbestos is suspected to be affecting the health of the residents at various sites from various locations. Due to this threat to human health, EPA has requested emergency environmental response support from the Volpe Center.

**SURROUNDING POPULATION:** ☐ Residential ☒ Industrial ☐ Rural ☒ Urban ☐ Other: \_\_\_\_\_

**HEALTH AND SAFETY PLAN FORM**

CDM FEDERAL PROGRAMS CORPORATION

CDM FPC Health and Safety Program

PROJECT DOCUMENT #: 2603-23-HSAP

Site Location Map

See Figures 1 and 2.





**HISTORY:**

The Zonolite Mine began operation in 1924 by owner Edward Alley. In 1925, Great Northern Railroad shipped the first boxcar of "Zonolite" from Libby to an Ohio company that used it to insulate bank vaults, office safes, and filing cabinets. Other firms used the material to make building boards and roofing materials. Processing the material was a straightforward process. The vermiculite ore was stripped from the mine and hauled in trucks to a mill, where it was separated into various commercial sizes through a screening system. Some of the ore was shipped untouched. Other material was sent to an expansion plant where it was run through ovens at about 2,000 degrees, causing it to expand to 15 times its original size. In 1939, Zonolite merged with another company mining at the bottom of the hill that eventually became known as the Zonolite Co. In 1963, the company was sold to W.R. Grace and Co. who expanded the operation and increased production. Through the 60s, 70s, and 80s, millions of tons of vermiculite ore was hauled by rail to Grace plants and other companies in 30 states and 6 foreign countries. At one time, 80 percent of the world's vermiculite came from Libby. The W.R. Grace Company, which owned the mine for 30 years, closed it in 1990 and sold the property four years later.

**WASTE TYPES:** ( ) Liquid (X) Solid ( ) Sludge ( ) Gas ( ) Unknown ( ) Other (Specify):

**WASTE CHARACTERISTICS:** Check as many as applicable.

- |               |               |                         |
|---------------|---------------|-------------------------|
| ( ) Corrosive | ( ) Flammable | ( ) Radioactive         |
| (X) Toxic     | ( ) Volatile  | ( ) Reactive            |
| ( ) Inert Gas | ( ) Unknown   | (X) Other: Carcinogenic |

**WORK ZONES:**

Work zones will be used during soil and vermiculite sampling and dust/air sampling. The exclusion zone will be soil sampling areas, interior of buildings, adjacent unpaved areas, and open piles of material (i.e., vermiculite) and waste. The contamination reduction zone will be demarcated by the decontamination station set up at each sampling site. The support zone will be considered the 10-foot perimeter around support vehicles.

**HAZARDS OF CONCERN:**

- |   |                             |
|---|-----------------------------|
| ( ) Heat Stress, attach guidelines          | ( ) Noise                   |
| (X) Cold Stress, attach guidelines          | ( ) Inorganic Chemicals     |
| ( ) Explosive/Flammable                     | ( ) Organic Chemicals       |
| ( ) Oxygen Deficient                        | (X) Motorized Traffic       |
| ( ) Radiological                            | (X) Heavy Machinery         |
| ( ) Biological:                             | (X) Slips, Trips, and Falls |
| (X) Other: Inhalation of particulate matter |                             |

**PRINCIPLE DISPOSAL METHODS AND PRACTICES:**

The sites (SLC1 and SLC2) associated with this investigation are currently owned by Mr. Scott Simmons and Utah Power & Light (Pacific Corp.), respectively. Both sites are where processing plants were located and where vermiculite ore was shipped. Potential problems include contaminated dust inside processing buildings and open piles of material and waste product resulting in exposure to workers onsite (SLC1), and possible product in shallow soils where the original processing plant was located (SLC2).

# HEALTH AND SAFETY PLAN FORM

CDM FEDERAL PROGRAMS CORPORATION

CDM FPC Health and Safety Program

PROJECT DOCUMENT #: 2603-23-HSAP

## HAZARDOUS MATERIAL SUMMARY: *Check waste type*

CHEMICALS: Amount/Units:	SOLIDS: Amounts/Units:	SLUDGES: Amounts/Units:	SOLVENTS: Amounts/Units:	OILS: Amounts/Units:	OTHER: Amounts/Units:
Acids	Flyash	Paint	Halogenated (chloro, bromo)	Oily Wastes	Laboratory
Pickling Liquors	✓ Asbestos	Pigments	Solvents	Gasoline	Pharmaceutical
Caustics	✓ Milling/Mine Tailings	Metal Sludges	Hydrocarbons	Diesel Oil	Hospital
Pesticides	Ferrous Smelter	POTW Sludge	Alcohols	Lubricants	Radiological
Dyes/Inks	Non-ferrous Smelter	Aluminum	Ketones	PCBs	Municipal
Cyanides	<u>Metals</u>	Distillation Bottoms	Esters	Polynuclear Aromatics	Construction
Phenols	Other: <u>Fe, Mn, Ni, Cd, Zn, Pb, Cu, Ag, Cr</u>	Other:	Esters	Other:	Munitions
Halogens			Other:		Other:
Dioxins					
Other:					

**OVERALL HAZARD EVALUATION:** ( ) High ( ) Medium (X) Low ( ) Unknown (Where tasks have different hazards, evaluate each. Attach additional sheets if necessary.)

**JUSTIFICATION:** CDM Federal personnel will avoid unnecessarily agitating suspect materials and visibly dusty conditions.

**FIRE/EXPLOSION POTENTIAL:** ( ) High ( ) Medium (X) Low ( ) Unknown

**BACKGROUND REVIEW:** ( ) Complete (X) Incomplete Additional information to be collected in future investigations.

## HEALTH AND SAFETY PLAN FORM

CDM FEDERAL PROGRAMS CORPORATION

CDM FPC Health and Safety Program

PROJECT DOCUMENT #: 2603-23-HSAP

KNOWN CONTAMINANTS	HIGHEST OBSERVED CONCENTRATION (specify units and media)	IDLH PEL/TLV ppm or mg/m <sup>3</sup> (aerosol)	IDLH ppm or mg/m <sup>3</sup> (aerosol)	WARNING CONCENTRATION ppm	SYMPTOMS/EFFECTS OF ACUTE EXPOSURE	PHOTOIONIZATION POTENTIAL
Asbestos	NA	NA	NA	NA	Assumed to be similar to overexposure of nuisance dust	
<div> <div> NA = Not Available S = Soil A = Air </div> <div> NE = None Established SW = Surface Water GW = Groundwater </div> <div> U = Unknown T = Tailings SL = Sludge </div> <div> OFF = Offsite W = Waste D = Drums </div> <div> TK = Tanks L = Lagoon SD = Sediment </div> </div>						



## HEALTH AND SAFETY PLAN FORM

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## FIELD ACTIVITIES COVERED UNDER THIS PLAN:

Task Description/Specific Technique-Standard Operating Procedures/Site Location (attach additional sheets as necessary)	TYPE	PRIMARY	CONTINGENCY	HAZARD SCHEDULE
1. Soil sampling (unpaved areas adjacent to process buildings).	<u>Intrusive</u>	<u>D</u> <u>Modified</u>	<u>C</u> <u>Exit Area</u>	<u>Low</u>
2. Process building dust/simulated disturbance with air sampling.	<u>Agitation</u>	<u>C</u> <u>Modified</u>	<u>Exit Area</u>	<u>Low</u>
3. Open piles of material (i.e., vermiculite) and waste	<u>Intrusive</u>	<u>C</u> <u>Modified</u>	<u>Exit Area</u>	<u>Low</u>
4. Ambient air monitoring	<u>Passive</u>	<u>D</u> <u>Modified</u>	<u>C</u> <u>Exit Area</u>	<u>Low</u>

## \* PERSONNEL AND RESPONSIBILITIES (include subcontractors)

NAME	FIRM/REGION	CDM FPC HEALTH CLEARANCE	RESPONSIBILITIES	ON SITE?
Tim Wall	CDM Federal/Cambridge	Yes	Project Manager	<u>1 - 2</u>
Frank Morris	CDM Federal/Denver	Yes	Site Health and Safety Coordinator	<u>1 - 2</u>
Melissa Petrack (Air)	Pacific Env. Services	Yes	Field Team Leader	<u>1 - 2</u>
Tommy Cook (Soil)	CDM Federal/Denver	Yes	Field Team Leader	<u>1 - 2</u>

## HEALTH AND SAFETY PLAN FORM

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Protective Equipment: *Specify by task. Indicate type and/or materials, as necessary. Use copies of this sheet if necessary.*

BLOCK A: TASK: 1,4 (X) PRIMARY  
LEVEL: D - MODIFIED ( ) CONTINGENCY

Respiratory: (X) Not Needed  
( ) SCBA, Airline: \_\_\_\_\_  
( ) APR: \_\_\_\_\_  
( ) Cartridge: \_\_\_\_\_  
( ) Escape Mask: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Head and Eye: ( ) Not Needed  
(X) Safety Glasses: \_\_\_\_\_  
( ) Face Shield: \_\_\_\_\_  
( ) Goggles: \_\_\_\_\_  
( ) Hard Hat: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Boots: ( ) Not Needed  
(X) Boots: Leather steel-toed work boots  
(X) Overboots: Rubber

Protective Clothing: ( ) Not Needed  
( ) Encapsulated Suit: \_\_\_\_\_  
( ) Splash Suit: \_\_\_\_\_  
( ) Apron: \_\_\_\_\_  
( ) Tyvek: \_\_\_\_\_  
( ) Saranex: \_\_\_\_\_  
(X) Coverall: Cloth  
( ) Other: \_\_\_\_\_

Gloves: ( ) Not Needed  
( ) Undergloves: \_\_\_\_\_  
(X) Gloves: Nitrile or Latex  
( ) Overgloves: \_\_\_\_\_  
( ) Other: Specify Below

BLOCK C: TASK: 1,2,3,4 ( ) PRIMARY  
LEVEL: Exit Area (X) CONTINGENCY

Respiratory: ( ) Not Needed  
( ) SCBA, Airline: \_\_\_\_\_  
( ) APR: Full or Half Face  
( ) Cartridge: P100  
( ) Escape Mask: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Head and Eye: ( ) Not Needed  
( ) Safety Glasses: \_\_\_\_\_  
( ) Face Shield: \_\_\_\_\_  
( ) Goggles: \_\_\_\_\_  
( ) Hard Hat: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Boots: ( ) Not Needed  
( ) Boots: Leather steel-toed work boots  
( ) Overboots: Tyvek booties

Protective Clothing: ( ) Not Needed  
( ) Encapsulated Suit: \_\_\_\_\_  
( ) Splash Suit: \_\_\_\_\_  
( ) Apron: \_\_\_\_\_  
( ) Tyvek: \_\_\_\_\_  
( ) Saranex: \_\_\_\_\_  
( ) Coverall: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Gloves: ( ) Not Needed  
( ) Undergloves: \_\_\_\_\_  
( ) Gloves: Nitrile or Latex  
( ) Overgloves: \_\_\_\_\_  
( ) Other: Specify Below

BLOCK B: TASK: 2,3 (X) PRIMARY  
LEVEL: C - MODIFIED ( ) CONTINGENCY

Respiratory: ( ) Not Needed  
( ) SCBA, Airline: \_\_\_\_\_  
(X) APR: Full or Half Face  
(X) Cartridge: P100  
( ) Escape Mask: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Head and Eye: ( ) Not Needed  
(X) Safety Glasses: \_\_\_\_\_  
( ) Face Shield: \_\_\_\_\_  
( ) Goggles: \_\_\_\_\_  
(X) Hard Hat: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Boots: ( ) Not Needed  
(X) Boots: Leather steel-toed work boots  
(X) Overboots: Rubber

Protective Clothing: ( ) Not Needed  
( ) Encapsulated Suit: \_\_\_\_\_  
( ) Splash Suit: \_\_\_\_\_  
( ) Apron: \_\_\_\_\_  
(X) Tyvek: \_\_\_\_\_  
( ) Saranex: \_\_\_\_\_  
( ) Coverall: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Gloves: ( ) Not Needed  
( ) Undergloves: \_\_\_\_\_  
(X) Gloves: Nitrile or Latex  
( ) Overgloves: \_\_\_\_\_  
( ) Other: Specify Below

BLOCK D: TASK: 2 ( ) PRIMARY  
LEVEL: LEAVE AREA (X) CONTINGENCY

Respiratory: ( ) Not Needed  
( ) SCBA, Airline: \_\_\_\_\_  
( ) APR: \_\_\_\_\_  
( ) Cartridge: \_\_\_\_\_  
( ) Escape Mask: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Head and Eye: ( ) Not Needed  
( ) Safety Glasses: \_\_\_\_\_  
( ) Face Shield: \_\_\_\_\_  
( ) Goggles: \_\_\_\_\_  
( ) Hard Hat: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Boots: ( ) Not Needed  
( ) Boots: \_\_\_\_\_  
( ) Overboots: \_\_\_\_\_

Protective Clothing: ( ) Not Needed  
( ) Encapsulated Suit: \_\_\_\_\_  
( ) Splash Suit: \_\_\_\_\_  
( ) Apron: \_\_\_\_\_  
( ) Tyvek: \_\_\_\_\_  
( ) Saranex: \_\_\_\_\_  
( ) Coverall: \_\_\_\_\_  
( ) Other: \_\_\_\_\_

Gloves: ( ) Not Needed  
( ) Undergloves: \_\_\_\_\_  
( ) Gloves: \_\_\_\_\_  
( ) Overgloves: \_\_\_\_\_  
( ) Other: Specify Below

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Monitoring Equipment: *Specify by task. Indicate type as necessary. Attach additional sheets as necessary.*

INSTRUMENT	TASK	ACTION GUIDELINES		COMMENTS (includes schedules of use)
Combustible Gas Indicator	1-2-3-4 (X) Not Needed			Entering tanks, vats, sumps, and other confined spaces is strictly forbidden.
Radiation Survey Meter	1-2-3-4-5-6 (X) Not Needed			Radiation is not an expected hazard.
Photoionization Detectors  ( ) 11.7 eV ( ) 10.2 eV ( ) 9.8 eV ( ) ____ eV  Type _____	1-2-3-4 (X) Not Needed	Specify:  Detectable odor	If odor of any kind is detected, cease work, move to fresh air.	If further work is necessary in the area where odors were detected, personnel protection will be evaluated.
Flame Ionization Detector Type _____	1-2-3-4 (X) Not Needed	Specify:  Detectable odor	If odor of any kind is detected, cease work, move to fresh air.	If further work is necessary in the area where odors were detected, personnel protection will be evaluated.
Detector Tubes/Monitor Type _____ Type _____	1-2-3-4 (X) Not Needed	Specify:		Toxic gases are not expected to be encountered. Entrance into confined spaces where toxic gases could be concentrated is strictly forbidden.
Respirable Dust Monitor Type <u>PDM-3 Miniram</u> Type _____	1-2-3-4 (X) Not Needed	Specify:	If team observes visible dust in air while working on or near the site they will cease work.	If dusty conditions persist, site will be abandoned and personnel protection reevaluated.
Other: Specify: Air Monitoring	1-2-3-4 ( ) Not Needed	Specify: NIOSH Methods	If team members notice eye or throat irritation, or other symptoms of exposure, they will cease work.	

**HEALTH AND SAFETY PLAN FORM**

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**DECONTAMINATION PROCEDURES:****PERSONALIZED DECONTAMINATION:**

Wash well before hand to mouth contact is employed. A shower will be taken as soon as possible after leaving the field. Workers will remove protective clothing in this order:

- 1) Wash overboots in soapy water and rinse
- 2) Remove overboots or booties
- 3) Remove gloves
- 4) Remove safety glasses
- 5) Remove Tyvek or cloth coverall, if used
- 6) Remove respirator, if used
- 7) Remove inner gloves
- 8) Wash hands/face before eating/drinking

( ) Not Needed

**SAMPLING EQUIPMENT DECONTAMINATION:**

See CDM Federal SOP 4-5. All sampling equipment will be thoroughly decontaminated as follows:

- 1) Wash and scrub with low phosphate detergent.
- 2) Potable tap water rinse.
- 3) Potable tap water rinse.
- 4) Thoroughly rinse with deionized water.
- 5) Air dry.
- 6) Wrap in aluminum foil for transport.

( ) Not Needed

**HEAVY EQUIPMENT DECONTAMINATION**

See CDM Federal SOP 4-5. All heavy equipment and tool parts that contact subsurface soil are constructed of heavy gauge steel and have no natural or synthetic components that could absorb and retain most soil-borne organic contaminants.

Prior to removal from the work site, potential contaminated soil/groundwater will be scraped or brushed from the exterior surfaces.

The drill rig, augers and any other large equipment in the exclusion zone will be taken to a decon pad and steam cleaned.

(X) Not Needed

**CONTAINMENT AND DISPOSAL METHOD:**

All disposable PPE will be double bagged prior to disposal. Decon water to be disposed onsite.

( ) Not Needed

**CONTAINMENT AND DISPOSAL METHOD:**

Decon water to be disposed onsite.

( ) Not Needed

**CONTAINMENT AND DISPOSAL METHOD:**

All derived liquids will be contained and held for appropriate disposal.

(X) Not Needed

**HEALTH AND SAFETY PLAN FORM**

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**EMERGENCY CONTACTS:**

Water Supply N/A  
Site Telephone N/A  
EPA Release Report #: 1-800-424-8802  
CDM 24-Hour Emergency: 1 571-216-7004  
Facility Management: N/A  
Other (specify):

**EMERGENCY CONTACTS:****NAME:****PHONE:**

Health and Safety Manager	Chuck Myers	(703) 968-0900
Project Manager	Tim Wall	(617) 452-6257
Site Safety Coordinator	Frank Morris	(720) 264-1119
DOT Contact	John McGuiggin	(617) 494-2574
EPA Contact	Joyce Ackerman	(303) 312-6822
Environmental Agency		(800) 234-5677
State Spill Number		911
Fire Department-Salt Lake City		911
Police Department-Salt Lake City		911
Highway Patrol		(801) 965-4505
Occupational Physical	Jerry Berke	1-800-350-4511
Poison Control Center		(801) 851-2151

**CONTINGENCY PLANS: Summarize below.**

Evacuate site if any unexpected hazardous conditions are encountered. If staff observe hazards for which they have not been prepared, they will withdraw from the area and call CDM Federal Health and Safety. CDM Federal personnel will leave the site and upgrade their level of protection if they experience nausea or dizziness. No volatile compounds are expected to be encountered at concentrations dangerous to human health. If any odors are noted, work will cease and personnel protection reevaluated. In the event of medical emergency, contact Hospital, Police, or Sheriff's Department. If respirable dust is noted, additional engineering controls will be implemented. If these controls do not eliminate the exposure, personnel protection will be reevaluated.

**MEDICAL EMERGENCY:**

Hospital: LDS Hospital

Hospital Address: 325 8<sup>th</sup> Avenue, Salt Lake City, UT 84143

Name of Contact at Hospital:

Name of 24-Hour Ambulance: Call 911

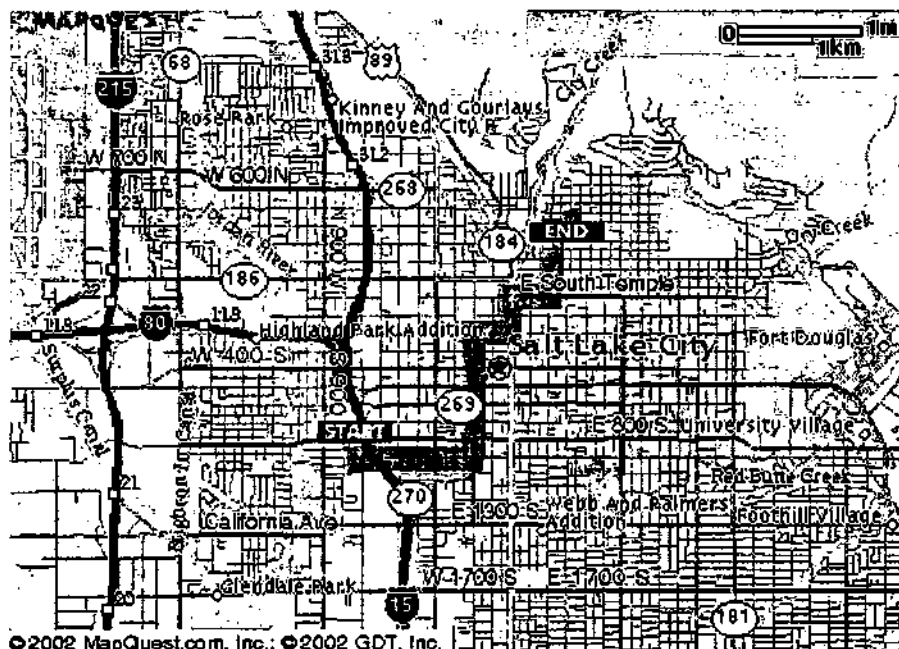
Route to Hospital: See Figures 3 and 4

**HEALTH AND SAFETY PLAN APPROVALS:**

Prepared by: Frank R. Morris Date: 7/14/03 *Modified Location*  
SHSC Signature: Chad J. Hyslop Date: 10/10/02  
HSM Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Route to hospital - See Figure 3

**FIGURE 3 – Route to LDS Hospital from SLC1**



733 W 800 S  
Salt Lake City, UT  
84104-1416 US

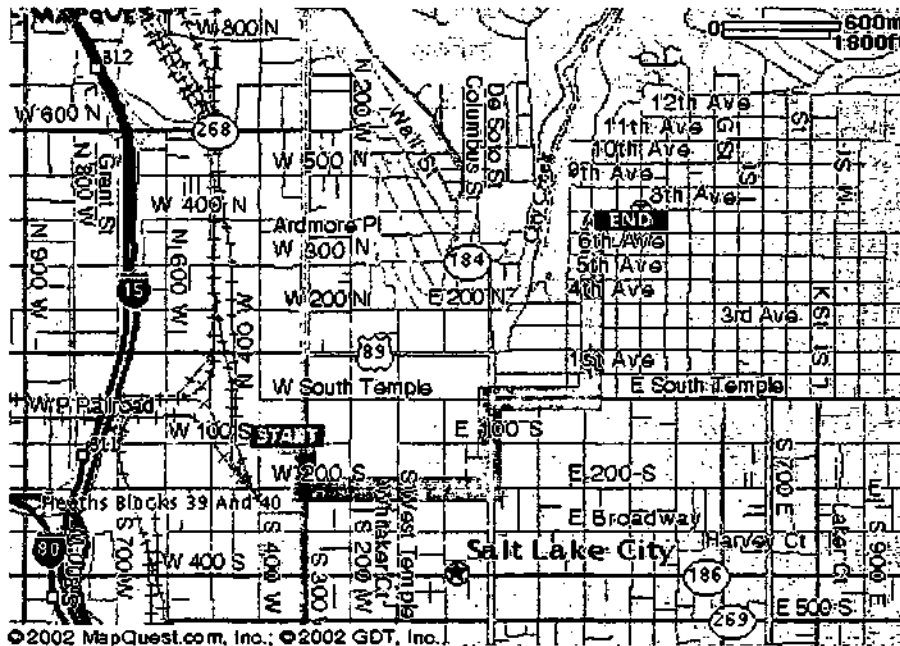
[300-348] 325 8th Ave  
Salt Lake City, UT  
84143-0001 US

**Total Distance: 3.83 miles**

**Total Estimated Time: 15 minutes**

DIRECTIONS	DISTANCE
1: Start out going East on W 800 S towards S 700 W by turning right.	0.05 miles
2: Turn RIGHT onto S 700 W.	0.14 miles
3: Turn LEFT onto W 900 S.	0.89 miles
4: Turn LEFT onto S WEST TEMPLE/UT-270.	0.75 miles
5: S WEST TEMPLE/UT-270 becomes S WEST TEMPLE.	0.29 miles
6: Turn RIGHT onto W 200 S.	0.29 miles
7: Turn LEFT onto S STATE ST/US-89.	0.30 miles
8: Turn RIGHT onto E SOUTH TEMPLE.	0.32 miles
9: Turn LEFT onto 8 ST.	0.55 miles
10: Turn RIGHT onto 7TH AVE.	0.15 miles
11: Turn LEFT onto D ST.	0.08 miles
<b>Total Estimated Time:</b> <b>15 minutes</b>	<b>Total Distance:</b> <b>3.83 miles</b>

**FIGURE 4 – Route to LDS Hospital from SLC2**



FROM	
333 W 100 S Salt Lake City, UT 84101-1209 US	[300-348] 325 8th Ave Salt Lake City, UT 84143-0001 US
Total Distance: 2.21 miles	Total Estimated Time: 9 minutes
DIRECTIONS	
1: Start out going East on W 100 S towards S 300 W/UT-186 by turning right.	0.06 miles
2: Turn RIGHT onto S 300 W/UT-186.	0.16 miles
3: Turn LEFT onto W 200 S.	0.60 miles
4: Turn LEFT onto S STATE ST/US-89.	0.30 miles
5: Turn RIGHT onto E SOUTH TEMPLE.	0.32 miles
6: Turn LEFT onto 8 ST.	0.55 miles
7: Turn RIGHT onto 7TH AVE.	0.15 miles
8: Turn LEFT onto D ST.	0.08 miles
Total Estimated Time: 9 minutes	Total Distance: 2.21 miles

The following personnel have read and fully understand the contents of this Health and Safety Plan and further agree to all requirements contained herein.



## HEALTH AND SAFETY PLAN FORM

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[illegible]

## **Appendix D**

### **Laboratory Analytical Method**

# ASBESTOS and OTHER FIBERS by PCM

7400

Various

MW: Various

CAS: Various

RTECS: Various

METHOD: 7400, Issue 2

EVALUATION: FULL

Issue 1: Rev. 3 on 15 May 1989

Issue 2: 15 August 1994

OSHA: 0.1 asbestos fiber (> 5 µm long)/cc;

1 f/cc/30 min excursion; carcinogen

MSHA: 2 asbestos fibers/cc

NIOSH: 0.1 f/cc (fibers > 5 µm long)/400 L; carcinogen

ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, fibers/cc; carcinogen

PROPERTIES: solid, fibrous, crystalline, anisotropic

SYNONYMS [CAS #]: actinolite [77536-66-4] or ferroactinolite [15669-07-5]; amosite [12172-73-5]; anthophyllite [77536-67-5]; chrysotile [12001-29-5]; serpentine [18786-24-8]; crocidolite [12001-28-4]; tremolite [77536-68-6]; amphibole asbestos [1332-21-4]; refractory ceramic fibers [142844-00-6]; fibrous glass.

SAMPLING		MEASUREMENT	
<b>SAMPLER:</b>	<b>FILTER</b> (0.45- to 1.2-µm cellulose ester membrane, 25-mm; conductive cowl on cassette)	<b>TECHNIQUE:</b>	LIGHT MICROSCOPY, PHASE CONTRAST
<b>FLOW RATE*:</b>	0.5 to 16 L/min	<b>ANALYTE:</b>	fibers (manual count)
<b>VOL-MIN*:</b>	400 L @ 0.1 fiber/cc	<b>SAMPLE PREPARATION:</b>	acetone - collapse/triacetin - immersion
<b>-MAX*:</b>	(step 4, sampling) *Adjust to give 100 to 1300 fiber/mm <sup>2</sup>	<b>COUNTING RULES:</b>	described in previous version of this method as "A" rules [1,3]
<b>SHIPMENT:</b>	routine (pack to reduce shock)	<b>EQUIPMENT:</b>	1. positive phase-contrast microscope 2. Walton-Beckett graticule (100-µm field of view) Type G-22 3. phase-shift test slide (HSE/NPL)
<b>SAMPLE STABILITY:</b>	stable	<b>CALIBRATION:</b>	HSE/NPL test slide
<b>BLANKS:</b>	2 to 10 field blanks per set	<b>RANGE:</b>	100 to 1300 fibers/mm <sup>2</sup> filter area
ACCURACY		<b>ESTIMATED LOD:</b>	7 fibers/mm <sup>2</sup> filter area
<b>RANGE STUDIED:</b>	80 to 100 fibers counted	<b>PRECISION (<math>\bar{S}_p</math>):</b>	0.10 to 0.12 [1]; see EVALUATION OF METHOD
<b>BIAS:</b>	See EVALUATION OF METHOD		
<b>OVERALL PRECISION (<math>\bar{S}_p</math>):</b>	0.115 to 0.13 [1]		
<b>ACCURACY:</b>	See EVALUATION OF METHOD		

**APPLICABILITY:** The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. The method gives an index of airborne fibers. It is primarily used for estimating asbestos concentrations, though PCM does not differentiate between asbestos and other fibers. Use this method in conjunction with electron microscopy (e.g., Method 7402) for assistance in identification of fibers. Fibers < ca. 0.25 µm diameter will not be detected by this method [4]. This method may be used for other materials such as fibrous glass by using alternate counting rules (see Appendix C).

**INTERFERENCES:** If the method is used to detect a specific type of fiber, any other airborne fiber may interfere since all particles meeting the counting criteria are counted. Chain-like particles may appear fibrous. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

**OTHER METHODS:** This revision replaces Method 7400, Revision #3 (date 5/15/89).

**REAGENTS:**

1. Acetone,\* reagent grade.
2. Triacetin (glycerol triacetate), reagent grade.

\* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically conductive extension cowl and cellulose ester filter, 0.45- to 1.2- $\mu$ m pore size, and backup pad.

NOTE 1: Analyze representative filters for fiber background before use to check for clarity and background. Discard the filter lot if mean is  $\geq 5$  fibers per 100 graticule fields. These are defined as laboratory blanks. Manufacturer-provided quality assurance checks on filter blanks are normally adequate as long as field blanks are analyzed as described below.

NOTE 2: The electrically conductive extension cowl reduces electrostatic effects. Ground the cowl when possible during sampling.

NOTE 3: Use 0.8- $\mu$ m pore size filters for personal sampling. The 0.45- $\mu$ m filters are recommended for sampling when performing TEM analysis on the same samples. However, their higher pressure drop precludes their use with personal sampling pumps.

NOTE 4: Other cassettes have been proposed that exhibit improved uniformity of fiber deposit on the filter surface, e.g., bellmouthed sampler (Envirometrics, Charleston, SC). These may be used if shown to give measured concentrations equivalent to sampler indicated above for the application.

2. Personal sampling pump, battery or line-powered vacuum, of sufficient capacity to meet flow-rate requirements (see step 4 for flow rate), with flexible connecting tubing.
3. Wire, multi-stranded, 22-gauge; 1", hose clamp to attach wire to cassette.
4. Tape, shrink- or adhesive-
5. Slides, glass, frosted-end, pre-cleaned, 25 x 75-mm.
6. Cover slips, 22- x 22-mm, No. 1-1/2, unless otherwise specified by microscope manufacturer.
7. Lacquer or nail polish.
8. Knife, #10 surgical steel, curved blade.
9. Tweezers.

#### EQUIPMENT:

10. Acetone flash vaporization system for clearing filters on glass slides (see ref. [5] for specifications or see manufacturer's instructions for equivalent devices).
11. Micropipets or syringes, 5- $\mu$ L and 100- to 500- $\mu$ L.
12. Microscope, positive phase (dark) contrast, with green or blue filter, adjustable field iris, 8 to 10X eyepiece, and 40 to 45X phase objective (total magnification ca. 400X); numerical aperture = 0.65 to 0.75.
13. Graticule, Walton-Beckett type with 100- $\mu$ m diameter circular field (area = 0.00785 mm<sup>2</sup>) at the specimen plane (Type G-22). Available from Optometrics USA, P.O. Box 699, Ayer, MA 01432 [phone (508)-772-1700], and McCrone Accessories and Components, 850 Pasquinelli Drive, Westmont, IL 60559 [phone (312) 887-7100].  
NOTE: The graticule is custom-made for each microscope. (see APPENDIX A for the custom-ordering procedure).
14. HSE/NPL phase contrast test slide, Mark II. Available from Optometrics USA (address above).
15. Telescope, ocular phase-ring centering.
16. Stage micrometer (0.01-mm divisions).

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**SPECIAL PRECAUTIONS:** Acetone is extremely flammable. Take precautions not to ignite it. Heating of acetone in volumes greater than 1 mL must be done in a ventilated laboratory fume hood using a flameless, spark-free heat source.

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#### SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. To reduce contamination and to hold the cassette tightly together, seal the crease between the cassette base and the cowl with a shrink band or light colored adhesive tape. For personal sampling, fasten the (uncapped) open-face cassette to the worker's lapel. The open face should be oriented downward.  
NOTE: The cowl should be electrically grounded during area sampling, especially under conditions of low relative humidity. Use a hose clamp to secure one end of the wire (Equipment, Item 3) to the monitor's cowl. Connect the other end to an earth ground (i.e., cold water pipe).
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Handle field blanks in a manner representative of actual handling of associated samples in the set. Open field blank cassettes at the same time as other cassettes just prior to sampling. Store top covers and cassettes in a clean area (e.g., a closed bag or box) with the top covers from the sampling cassettes during the sampling period.
4. Sample at 0.5 L/min or greater [6]. Adjust sampling flow rate,  $Q$  (L/min), and time,  $t$  (min), to produce a fiber density,  $E$ , of 100 to 1300 fibers/mm<sup>2</sup> ( $3.85 \cdot 10^4$  to  $5 \cdot 10^5$  fibers per 25-mm filter with effective collection area  $A_c = 385$  mm<sup>2</sup>) for optimum accuracy. These variables are related to the action level (one-half the current standard),  $L$  (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}, \text{ min.}$$

NOTE 1: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. The collection efficiency does not appear to be a function of flow rate in the range of 0.5 to 16 L/min for asbestos fibers [7]. Relatively large diameter fibers ( $>3 \mu\text{m}$ ) may exhibit significant aspiration loss and inlet deposition. A sampling rate of 1 to 4 L/min for 8 h is appropriate in atmospheres containing ca. 0.1 fiber/cc in the absence of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes ( $\leq 400 \text{ L}$ ) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If  $\geq 50\%$  of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration.

NOTE 2: OSHA regulations specify a minimum sampling volume of 48 L for an excursion measurement, and a maximum sampling rate of 2.5 L/min [3].

5. At the end of sampling, replace top cover and end plugs.
6. Ship samples with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.

NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

#### SAMPLE PREPARATION:

NOTE 1: The object is to produce samples with a smooth (non-grainy) background in a medium with refractive index  $\leq 1.46$ . This method collapses the filter for easier focusing and produces permanent (1 - 10 years) mounts which are useful for quality control and interlaboratory comparison. The aluminum "hot block" or similar flash vaporization techniques may be used outside the laboratory [2]. Other mounting techniques meeting the above criteria may also be used (e.g., the laboratory fume hood procedure for generating acetone vapor as described in Method 7400 - revision of 5/15/85, or the non-permanent field mounting technique used in P&CAM 239 [3,7,8,9]). Unless the effective filtration area is known, determine the area and record the information referenced against the sample ID number [1,9,10,11].

NOTE 2: Excessive water in the acetone may slow the clearing of the filter, causing material to be washed off the surface of the filter. Also, filters that have been exposed to high humidities prior to clearing may have a grainy background.

7. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Adjust the rheostat to heat the "hot block" to ca.  $70^\circ\text{C}$  [2].  
NOTE: If the "hot block" is not used in a fume hood, it must rest on a ceramic plate and be isolated from any surface susceptible to heat damage.
9. Mount a wedge cut from the sample filter on a clean glass slide.
  - a. Cut wedges of ca. 25% of the filter area with a curved-blade surgical steel knife using a rocking motion to prevent tearing. Place wedge, dust side up, on slide.  
NOTE: Static electricity will usually keep the wedge on the slide.

- b. Insert slide with wedge into the receiving slot at base of "hot block". Immediately place tip of a micropipet containing ca. 250  $\mu$ L acetone (use the minimum volume needed to consistently clear the filter sections) into the inlet port of the PTFE cap on top of the "hot block" and inject the acetone into the vaporization chamber with a slow, steady pressure on the plunger button while holding pipet firmly in place. After waiting 3 to 5 sec for the filter to clear, remove pipet and slide from their ports.

CAUTION: Although the volume of acetone used is small, use safety precautions. Work in a well-ventilated area (e.g., laboratory fume hood). Take care not to ignite the acetone. Continuous use of this device in an unventilated space may produce explosive acetone vapor concentrations.

- c. Using the 5- $\mu$ L micropipet, immediately place 3.0 to 3.5  $\mu$ L triacetin on the wedge. Gently lower a clean cover slip onto the wedge at a slight angle to reduce bubble formation. Avoid excess pressure and movement of the cover glass.

NOTE: If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excessive triacetin remains at the edge of the filter under the cover slip, fiber migration may occur.

- d. Mark the outline of the filter segment with a glass marking pen to aid in microscopic evaluation.
- e. Glue the edges of the cover slip to the slide using lacquer or nail polish [12]. Counting may proceed immediately after clearing and mounting are completed.

NOTE: If clearing is slow, warm the slide on a hotplate (surface temperature 50 °C) for up to 15 min to hasten clearing. Heat carefully to prevent gas bubble formation.

#### CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturers instructions. At least once daily use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric. With each microscope, keep a logbook in which to record the dates of microscope cleanings and major servicing.
  - a. Each time a sample is examined, do the following:
    - (1) Adjust the light source for even illumination across the field of view at the condenser iris. Use Kohler illumination, if available. With some microscopes, the illumination may have to be set up with bright field optics rather than phase contract optics.
    - (2) Focus on the particulate material to be examined.
    - (3) Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.
  - b. Check the phase-shift detection limit of the microscope periodically for each analyst/microscope combination:
    - (1) Center the HSE/NPL phase-contrast test slide under the phase objective.
    - (2) Bring the blocks of grooved lines into focus in the graticule area.
 

NOTE: The slide contains seven blocks of grooves (ca. 20 grooves per block) in descending order of visibility. For asbestos counting the microscope optics must completely resolve the grooved lines in block 3 although they may appear somewhat faint, and the grooved lines in blocks 6 and 7 must be invisible when centered in the graticule area. Blocks 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has resolution either too low or too high for fiber counting.
    - (3) If image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.
11. Document the laboratory's precision for each counter for replicate fiber counts.
  - a. Maintain as part of the laboratory quality assurance program a set of reference slides to be used on a daily basis [13]. These slides should consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field and reference samples (e.g., PAT, AAR, commercial samples). The Quality Assurance Officer

should maintain custody of the reference slides and should supply each counter with a minimum of one reference slide per workday. Change the labels on the reference slides periodically so that the counter does not become familiar with the samples.

- b. From blind repeat counts on reference slides, estimate the laboratory intra- and intercounter precision. Obtain separate values of relative standard deviation ( $S_r$ ) for each sample matrix analyzed in each of the following ranges: 5 to 20 fibers in 100 graticule fields, >20 to 50 fibers in 100 graticule fields, and >50 to 100 fibers in 100 graticule fields. Maintain control charts for each of these data files.

NOTE: Certain sample matrices (e.g., asbestos cement) have been shown to give poor precision [9].

12. Prepare and count field blanks along with the field samples. Report counts on each field blank.

NOTE 1: The identity of blank filters should be unknown to the counter until all counts have been completed.

NOTE 2: If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

13. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter). Use the following test to determine whether a pair of counts by the same counter on the same filter should be rejected because of possible bias: Discard the sample if the absolute value of the difference between the square roots of the two counts (in fiber/mm<sup>2</sup>) exceeds  $2.77 (X)S_r$ , where  $X$  = average of the square roots of the two fiber counts

(in fiber/mm<sup>2</sup>) and  $S_r = \frac{S_r}{2}$ , where  $S_r$  is the intracounter relative standard deviation for the

appropriate count range (in fibers) determined in step 11. For more complete discussions see reference [13].

NOTE 1: Since fiber counting is the measurement of randomly placed fibers which may be described by a Poisson distribution, a square root transformation of the fiber count data will result in approximately normally distributed data [13].

NOTE 2: If a pair of counts is rejected by this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts. It is not necessary to use this statistic on blank counts.

14. The analyst is a critical part of this analytical procedure. Care must be taken to provide a non-stressful and comfortable environment for fiber counting. An ergonomically designed chair should be used, with the microscope eyepiece situated at a comfortable height for viewing. External lighting should be set at a level similar to the illumination level in the microscope to reduce eye fatigue. In addition, counters should take 10-to-20 minute breaks from the microscope every one or two hours to limit fatigue [14]. During these breaks, both eye and upper back/neck exercises should be performed to relieve strain.
15. All laboratories engaged in asbestos counting should participate in a proficiency testing program such as the AIHA-NIOSH Proficiency Analytical Testing (PAT) Program for asbestos and routinely exchange field samples with other laboratories to compare performance of counters.

## MEASUREMENT:

16. Center the slide on the stage of the calibrated microscope under the objective lens. Focus the microscope on the plane of the filter.

17. Adjust the microscope (Step 10).

NOTE: Calibration with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25  $\mu$ m) [4].

18. Counting rules: (same as P&CAM 239 rules [1,10,11]; see examples in APPENDIX B).

- a. Count any fiber longer than 5  $\mu$ m which lies entirely within the graticule area.

(1) Count only fibers longer than 5  $\mu$ m. Measure length of curved fibers along the curve.

(2) Count only fibers with a length-to-width ratio equal to or greater than 3:1.

- b. For fibers which cross the boundary of the graticule field:

(1) Count as 1/2 fiber any fiber with only one end lying within the graticule area, provided that the fiber meets the criteria of rule a above.



- (2) Do not count any fiber which crosses the graticule boundary more than once.
  - (3) Reject and do not count all other fibers.
  - c. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
  - d. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count.
19. Start counting from the tip of the filter wedge and progress along a radial line to the outer edge. Shift up or down on the filter, and continue in the reverse direction. Select graticule fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. Ensure that, as a minimum, each analysis covers one radial line from the filter center to the outer edge of the filter. When an agglomerate or bubble covers ca. 1/6 or more of the graticule field, reject the graticule field and select another. Do not report rejected graticule fields in the total number counted.
- NOTE 1: When counting a graticule field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count. A minimum counting time of 15 seconds per field is appropriate for accurate counting.
- NOTE 2: This method does not allow for differentiation of fibers based on morphology. Although some experienced counters are capable of selectively counting only fibers which appear to be asbestiform, there is presently no accepted method for ensuring uniformity of judgment between laboratories. It is, therefore, incumbent upon all laboratories using this method to report total fiber counts. If serious contamination from non-asbestos fibers occurs in samples, other techniques such as transmission electron microscopy must be used to identify the asbestos fiber fraction present in the sample (see NIOSH Method 7402). In some cases (i.e., for fibers with diameters  $>1\ \mu\text{m}$ ), polarized light microscopy (as in NIOSH Method 7403) may be used to identify and eliminate interfering non-crystalline fibers [15].
- NOTE 3: Do not count at edges where filter was cut. Move in at least 1 mm from the edge.
- NOTE 4: Under certain conditions, electrostatic charge may affect the sampling of fibers. These electrostatic effects are most likely to occur when the relative humidity is low (below 20%), and when sampling is performed near the source of aerosol. The result is that deposition of fibers on the filter is reduced, especially near the edge of the filter. If such a pattern is noted during fiber counting, choose fields as close to the center of the filter as possible [5].
- NOTE 5: Counts are to be recorded on a data sheet that provides, as a minimum, spaces on which to record the counts for each field, filter identification number, analyst's name, date, total fibers counted, total fields counted, average count, fiber density, and commentary. Average count is calculated by dividing the total fiber count by the number of fields observed. Fiber density (fibers/ $\text{mm}^2$ ) is defined as the average count (fibers/field) divided by the field (graticule) area ( $\text{mm}^2/\text{field}$ ).

## CALCULATIONS AND REPORTING OF RESULTS

20. Calculate and report fiber density on the filter,  $E$  (fibers/ $\text{mm}^2$ ), by dividing the average fiber count per graticule field,  $F/n_f$ , minus the mean field blank count per graticule field,  $B/n_b$ , by the graticule field area,  $A_f$  (approx.  $0.00785\ \text{mm}^2$ ):

$$E = \frac{\left( \frac{F}{n_f} - \frac{B}{n_b} \right)}{A_f}, \text{ fibers/mm}^2.$$

NOTE: Fiber counts above 1300 fibers/mm<sup>2</sup> and fiber counts from samples with >50% of filter area covered with particulate should be reported as "uncountable" or "probably biased." Other fiber counts outside the 100-1300 fiber/mm<sup>2</sup> range should be reported as having "greater than optimal variability" and as being "probably biased."

21. Calculate and report the concentration, C (fibers/cc), of fibers in the air volume sampled, V (L), using the effective collection area of the filter, A<sub>c</sub> (approx. 385 mm<sup>2</sup> for a 25-mm filter):

$$C = \frac{(E)(A_c)}{V \cdot 10^3}$$

NOTE: Periodically check and adjust the value of A<sub>c</sub> if necessary.

22. Report intralaboratory and interlaboratory relative standard deviations (from Step 11) with each set of results.

NOTE: Precision depends on the total number of fibers counted [1,16]. Relative standard deviation is documented in references [1,15-17] for fiber counts up to 100 fibers in 100 graticule fields. Comparability of interlaboratory results is discussed below. As a first approximation, use 213% above and 49% below the count as the upper and lower confidence limits for fiber counts greater than 20 (Fig. 1).

#### EVALUATION OF METHOD:

- A. This method is a revision of P&CAM 239 [10]. A summary of the revisions is as follows:

1. Sampling:

The change from a 37-mm to a 25-mm filter improves sensitivity for similar air volumes. The change in flow rates allows for 2-m<sup>3</sup> full-shift samples to be taken, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not a function of flow rate in the range 0.5 to 16 L/min [10].

2. Sample Preparation Technique:

The acetone vapor-triacetin preparation technique is a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [2,4,10]. The aluminum "hot block" technique minimizes the amount of acetone needed to prepare each sample.

3. Measurement:

- a. The Walton-Beckett graticule standardizes the area observed [14,18,19].
- b. The HSE/NPL test slide standardizes microscope optics for sensitivity to fiber diameter [4,14].
- c. Because of past inaccuracies associated with low fiber counts, the minimum recommended loading has been increased to 100 fibers/mm<sup>2</sup> filter area (a total of 78.5 fibers counted in 100 fields, each with field area = .00785 mm<sup>2</sup>.) Lower levels generally result in an overestimate of the fiber count when compared to results in the recommended analytical range [20]. The recommended loadings should yield intracounter S<sub>r</sub> in the range of 0.10 to 0.17 [21,22,23].

- B. Interlaboratory comparability:

An international collaborative study involved 16 laboratories using prepared slides from the asbestos cement, milling, mining, textile, and friction material industries [9]. The relative standard deviations (S<sub>r</sub>) varied with sample type and laboratory. The ranges were:

	<u>Intralaboratory S<sub>r</sub></u>	<u>Interlaboratory S<sub>r</sub></u>	<u>Overall S<sub>r</sub></u>
AIA (NIOSH A Rules)*	0.12 to 0.40	0.27 to 0.85	0.46
Modified CRS (NIOSH B Rules)**	0.11 to 0.29	0.20 to 0.35	0.25

\* Under AIA rules, only fibers having a diameter less than 3 µm are counted and fibers attached to particles larger than 3 µm are not counted. NIOSH A Rules are otherwise similar to the AIA rules.

\*\* See Appendix C.

A NIOSH study conducted using field samples of asbestos gave intralaboratory S<sub>r</sub> in the range 0.17 to 0.25 and an interlaboratory S<sub>r</sub> of 0.45 [21]. This agrees well with other recent studies [9,14,16].

At this time, there is no independent means for assessing the overall accuracy of this method. One measure of reliability is to estimate how well the count for a single sample agrees with the mean count from a large number of laboratories. The following discussion indicates how this estimation can be carried out based on measurements of the interlaboratory variability, as well as showing how the results of this method relate to the theoretically attainable counting precision and to measured intra- and interlaboratory S<sub>r</sub>. (NOTE: The following discussion does not include bias estimates and should not be taken to indicate that lightly loaded samples are as accurate as properly loaded ones).

Theoretically, the process of counting randomly (Poisson) distributed fibers on a filter surface will give an S<sub>r</sub> that depends on the number, N, of fibers counted:

$$S_r = 1/(N)^{1/2} \quad (1)$$

Thus S<sub>r</sub> is 0.1 for 100 fibers and 0.32 for 10 fibers counted. The actual S<sub>r</sub> found in a number of studies is greater than these theoretical numbers [17,19,20,21].

An additional component of variability comes primarily from subjective interlaboratory differences. In a study of ten counters in a continuing sample exchange program, Ogden [15] found this subjective component of intralaboratory S<sub>r</sub> to be approximately 0.2 and estimated the overall S<sub>r</sub> by the term:

$$\frac{[N + (0.2 \cdot N)^2]^{1/2}}{N} \quad (2)$$

Ogden found that the 90% confidence interval of the individual intralaboratory counts in relation to the means were +2 S<sub>r</sub> and - 1.5 S<sub>r</sub>. In this program, one sample out of ten was a quality control sample. For laboratories not engaged in an intensive quality assurance program, the subjective component of variability can be higher.

In a study of field sample results in 46 laboratories, the Asbestos Information Association also found that the variability had both a constant component and one that depended on the fiber count [14]. These results gave a subjective interlaboratory component of S<sub>r</sub> (on the same basis as Ogden's) for field samples of ca. 0.45. A similar value was obtained for 12 laboratories analyzing a set of 24 field samples [21]. This value falls slightly above the range of S<sub>r</sub> (0.25 to 0.42 for 1984-85) found for 80 reference laboratories in the NIOSH PAT program for laboratory-generated samples [17].

A number of factors influence S<sub>r</sub> for a given laboratory, such as that laboratory's actual counting performance and the type of samples being analyzed. In the absence of other information, such as from an interlaboratory quality assurance program using field samples, the value for the subjective component of variability is chosen as 0.45. It is hoped that the laboratories will carry out the recommended interlaboratory quality assurance programs to improve their performance and thus reduce the S<sub>r</sub>.

The above relative standard deviations apply when the population mean has been determined. It is more useful, however, for laboratories to estimate the 90% confidence interval on the mean count from a single sample fiber count (Figure 1). These curves assume similar shapes of the count distribution for interlaboratory and intralaboratory results [16].

For example, if a sample yields a count of 24 fibers, Figure 1 indicates that the mean interlaboratory count will fall within the range of 227% above and 52% below that value 90% of the time. We can apply these percentages directly to the air concentrations as well. If, for instance, this sample (24 fibers counted) represented a 500-L volume, then the measured concentration is 0.02 fibers/mL (assuming 100 fields counted, 25-mm filter, 0.00785 mm<sup>2</sup> counting field area). If this same sample were counted by a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.08 fiber/mL. These limits should be reported in any comparison of results between laboratories.

Note that the  $S_r$  of 0.45 used to derive Figure 1 is used as an estimate for a random group of laboratories. If several laboratories belonging to a quality assurance group can show that their interlaboratory  $S_r$  is smaller, then it is more correct to use that smaller  $S_r$ . However, the estimated  $S_r$  of 0.45 is to be used in the absence of such information. Note also that it has been found that  $S_r$  can be higher for certain types of samples, such as asbestos cement [9].

Quite often the estimated airborne concentration from an asbestos analysis is used to compare to a regulatory standard. For instance, if one is trying to show compliance with an 0.5 fiber/mL standard using a single sample on which 100 fibers have been counted, then Figure 1 indicates that the 0.5 fiber/mL standard must be 213% higher than the measured air concentration. This indicates that if one measures a fiber concentration of 0.16 fiber/mL (100 fibers counted), then the mean fiber count by a group of laboratories (of which the compliance laboratory might be one) has a 95% chance of being less than 0.5 fibers/mL; i.e.,  $0.16 + 2.13 \times 0.16 = 0.5$ .

It can be seen from Figure 1 that the Poisson component of the variability is not very important unless the number of fibers counted is small. Therefore, a further approximation is to simply use +213% and -49% as the upper and lower confidence values of the mean for a 100-fiber count.

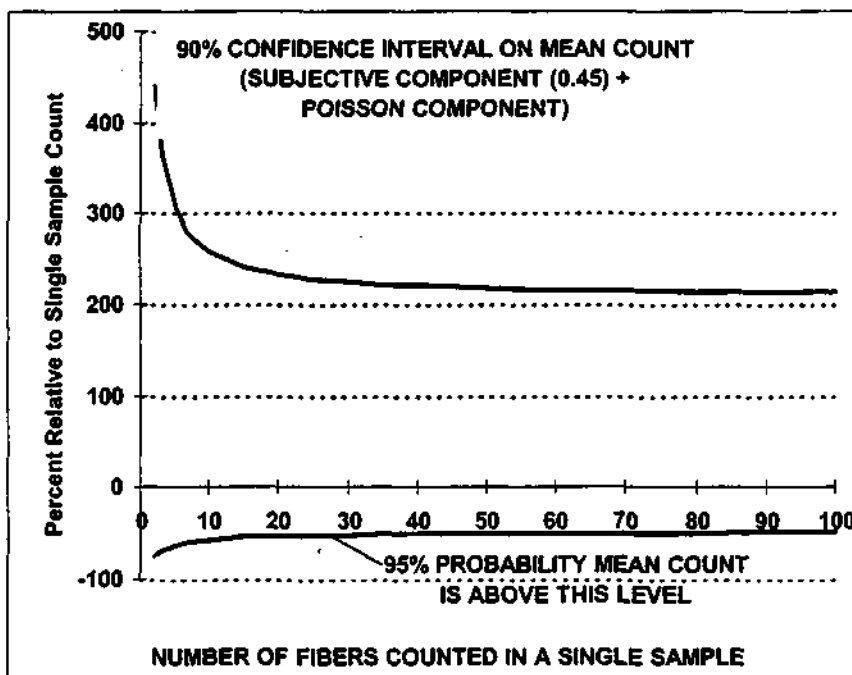


Figure 1. Interlaboratory Precision of Fiber Counts

The curves in Figures 1 are defined by the following equations:

$$\text{UCL} = \frac{2X + 2.25 + [(2.25 + 2X)^2 - 4(1 - 2.25S_r^2)X^2]^{1/2}}{2(1 - 2.25S_r^2)} \quad (3)$$

$$\text{LCL} = \frac{2X + 4 - [(4 + 2X)^2 - 4(1 - 4S_r^2)X^2]^{1/2}}{2(1 - 4S_r^2)} \quad (4)$$

where  $S_r$  = subjective interlaboratory relative standard deviation, which is close to the total interlaboratory  $S$ , when approximately 100 fibers are counted.

$X$  = total fibers counted on sample

LCL = lower 95% confidence limit.

UCL = upper 95% confidence limit.

Note that the range between these two limits represents 90% of the total range.

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#### APPENDIX A: CALIBRATION OF THE WALTON-BECKETT GRATICULE:

Before ordering the Walton-Beckett graticule, the following calibration must be done to obtain a counting area (D) 100  $\mu\text{m}$  in diameter at the image plane. The diameter,  $d_c$  (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

1. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
2. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.
3. Install the 40 to 45X phase objective.
4. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
5. Measure the magnified grid length of the graticule,  $L_g$  ( $\mu\text{m}$ ), using the stage micrometer.
6. Remove the graticule from the microscope and measure its actual grid length,  $L_a$  (mm). This can best be accomplished by using a stage fitted with verniers.
7. Calculate the circle diameter,  $d_c$  (mm), for the Walton-Beckett graticule:



These rules are sometimes referred to as the "A" rules.

### FIBER COUNT

<u>Object</u>	<u>Count</u>	<u>DISCUSSION</u>
1	1 fiber	Optically observable asbestos fibers are actually bundles of fine fibrils. If the fibrils seem to be from the same bundle the object is counted as a single fiber. Note, however, that all objects meeting length and aspect ratio criteria are counted whether or not they appear to be asbestos.
2	2 fiber	If fibers meeting the length and aspect ratio criteria (length $>5\ \mu\text{m}$ and length-to-width ratio $>3$ to 1) overlap, but do not seem to be part of the same bundle, they are counted as separate fibers.
3	1 fiber	Although the object has a relatively large diameter ( $>3\ \mu\text{m}$ ), it is counted as fiber under the rules. There is no upper limit on the fiber diameter in the counting rules. Note that fiber width is measured at the widest compact section of the object.
4	1 fiber	Although long fine fibrils may extend from the body of a fiber, these fibrils are considered part of the fiber if they seem to have originally been part of the bundle.
5	Do not count	If the object is $\leq 5\ \mu\text{m}$ long, it is not counted.
6	1 fiber	A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and aspect ratio criteria, they are counted as separate fibers.
7	1/2 fiber	A fiber which crosses into the graticule area one time is counted as 1/2 fiber.
8	Do not count	Ignore fibers that cross the graticulate boundary more than once.
9	Do not count	Ignore fibers that lie outside the graticule boundary.



**APPENDIX C. ALTERNATE COUNTING RULES FOR NON-ASBESTOS FIBERS**

Other counting rules may be more appropriate for measurement of specific non-asbestos fiber types, such as fibrous glass. These include the "B" rules given below (from NIOSH Method 7400, Revision #2, dated 8/15/87), the World Health Organization reference method for man-made mineral fiber [24], and the NIOSH fibrous glass criteria document method [25]. The upper diameter limit in these methods prevents measurements of non-thoracic fibers. It is important to note that the aspect ratio limits included in these methods vary. NIOSH recommends the use of the 3:1 aspect ratio in counting fibers.

It is emphasized that hybridization of different sets of counting rules is not permitted. Report specifically which set of counting rules are used with the analytical results.

**"B" COUNTING RULES:**

1. Count only ends of fibers. Each fiber must be longer than 5  $\mu\text{m}$  and less than 3  $\mu\text{m}$  diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules 1 and 2 above. Add split ends to the count as appropriate if the split fiber segment also meets the criteria of rules 1 and 2 above.
4. Count visibly free ends which meet rules 1 and 2 above when the fiber appears to be attached to another particle, regardless of the size of the other particle. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than 3  $\mu\text{m}$  in diameter.
5. Count free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules 1 and 2 above.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 graticule fields. Stop at 100 graticule fields, regardless of count.
7. Divide total end count by 2 to yield fiber count.

**APPENDIX D. EQUIVALENT LIMITS OF DETECTION AND QUANTITATION**

<u>fiber density on filter*</u>		<u>fiber concentration in air, f/cc</u>	
<u>fibers</u>		<u>400-L air</u>	<u>1000-L air</u>
<u>per 100 fields</u>	<u>fibers/mm<sup>2</sup></u>	<u>sample</u>	<u>sample</u>
200	255	0.25	0.10
100	127	0.125	0.05
LOQ 80	102	0.10	0.04
50	64	0.0625	0.025
25	32	0.03	0.0125
20	25	0.025	0.010
10	12.7	0.0125	0.005
8	10.2	0.010	0.004
LOD 5.5	7	0.00675	0.0027

\* Assumes 385 mm<sup>2</sup> effective filter collection area, and field area = 0.00785 mm<sup>2</sup>, for relatively "clean" (little particulate aside from fibers) filters.

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**ANALYSIS OF SOIL-LIKE MEDIA FOR ASBESTOS BY POLARIZED LIGHT MICROSCOPY**

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Date: March 3, 2003

SOP No. SRC-LIBBY-03 (Revision 0)

Title: **ANALYSIS OF ASBESTOS FIBERS IN SOIL BY POLARIZED LIGHT MICROSCOPY**

Author: William Brattin

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**SYNOPSIS:** A semi-quantitative method for identifying and quantifying asbestos fibers in soil using polarized light microscopy (PLM) is provided. This method is based on NIOSH Method 9002, EPA Method 600/R-93/116, and CARB Method 435, with project-specific modifications intended specifically for use at the Libby Superfund Site.

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**APPROVALS:**

TEAM MEMBER	SIGNATURE/TITLE	DATE
USEPA Region 8	<u>N/A</u>	<u>EDW 7/21/03</u>
Syracuse Research Corp.	<u>"</u>	<u>"</u>

Revision	Date	Principal Changes
0	03/03/03	--

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## **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide a standard approach for semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using polarized light microscopy (PLM). This SOP is specifically intended for application at the Libby Superfund site.

## **2.0 SCOPE AND APPLICATION**

This method is intended mainly for analysis of asbestos in soil or other similar soil-like media. This method is appropriate for the analysis of all types of asbestos fibers, including both chrysotile and amphiboles, including those that are characteristic of the Libby site.

## **3.0 RESPONSIBILITIES**

It is the responsibility of the laboratory supervisor to ensure that all analyses and quality assurance procedures are performed in accord with this SOP, and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation or analysis. The laboratory supervisor should also communicate with project managers at EPA or their oversight contractors any situations where a change from the SOP may be useful, and must receive approval from EPA for any deviation or modification from the SOP before proceeding with sample preparation and analysis.

## **4.0 METHOD DESCRIPTION**

The soil sample to be evaluated for asbestos content by PLM is examined under stereomicroscopy and under PLM (3-5 slides per sample) to estimate the amount of asbestos present. Quantification of the amount of asbestos present may be done either using a visual estimation approach or by a point counting approach, as specified in the Chain of Custody request. In either case, the concentration of Libby amphibole asbestos in the sample is estimated in terms of mass fraction (i.e., percent asbestos by weight) based on the use of project-specific reference materials.

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## 5.0 DETAILED METHOD

### 5.1 Basic Methods

All qualitative and quantitative analyses are to be performed in general accordance with the methods and techniques specified in NIOSH 9002, EPA 600/R-93/116, and CARB Method 435. Project-specific modification, clarifications, and requirements are provided below.

### 5.2 Visual Estimation Approach

#### 5.2.1 *Classification of Asbestos Mineral Type*

Based on fiber attributes (morphology, refractive index, color, birefringence, etc.), asbestos in the sample is classified into one of three categories:

Code	Description	Notes
LA	Libby Amphibole	Refractive index values for LA span the standard values for tremolite/actinolite (EPA 1993), but may include values for other similar amphiboles (e.g., winchite, richterite) characteristic of the mine at Libby. Based on analysis of 4 different samples from the mine (Wylie and Verkouteren 2000; USGS, unpublished data; Verkouteren, personal communication), observed refractive indices of Libby amphiboles range from about 1.629-1.640 $\gamma$ and 1.614-1.623 $\alpha$ , with a birefringence of about 0.017. The full range of refractive indices of samples from the mine may be somewhat greater.
OA	Other amphibole	Includes amphibole forms (e.g., amosite, crocidolite, anthophyllite) that are not thought to occur in significant amount at the mine in Libby
C	Chrysotile	

#### 5.2.2 *Estimation of LA Mass Percent*

The visual area estimation is a semi-quantitative approach that requires the microscopist to estimate the area fraction of the total material present in a field of view that consists of asbestos material. Because this estimation may be difficult, especially at low concentration values, and

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because the desired output is an estimate of mass fraction (rather than area fraction), all visual estimates of Libby amphibole content will be performed using a set of site-specific reference materials as a frame of reference. These reference material will contain either 0.2 % or 1.0% Libby amphibole by weight, and have been prepared for analysis using the same approach as for field samples. Because the ability to visualize asbestos under stereomicroscopy and PLM may depend in part on the attributes of the soil matrix, reference materials have been prepared using two different site-specific soils, referred to as "brown" and "tan". These two soil types are generally similar in their principal mineral components, but may differ in the relative amounts of trace mineral components and in their organic content.

The microscopist will examine the field soil and determine which type of reference material (tan or brown) most closely resembles the field sample. (If the field sample is substantially different in appearance than either type of reference material, this should be noted in the sample results). Then, using the two reference concentrations (0.2% and 1.0% ) of that soil type as a visual guide, the microscopist will evaluate the field sample and report the results as follows:

PLM Laboratory Report			Description
Qual	Conc (wt.%)	Bin	
ND		A	Asbestos was not observed in the field sample
Tr		B1	Asbestos was observed in the field sample at a level that appeared to be lower than the 0.2% reference material
<	1	B2	Asbestos was observed in the field sample at a level that appeared to exceed the 0.2% reference material but was less than the 1% reference material.
	1, 2, 3, etc	C	Asbestos was observed in the field sample at a level that appeared to equal or exceed the 1% standard. In this case, the mass percent is estimated quantitatively.

"ND" (not detected) in the Qualifier column is used for all samples in which asbestos is not observed under stereomicroscopy and is also not detected in five (5) different PLM slides prepared using representative sub-samples of the test material. These samples are assigned to Bin A.

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**"Tr" (trace) in the Qualifier column** is used for all samples in which asbestos is observed either under stereomicroscopy or in at least one out of 3-5 PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be less than the 0.2 % reference material (tan and/or brown, whichever is most similar in appearance to the test material). These samples are assigned to **Bin B1**.

**"<" (less than) in the Qualifier column and 1 in the Concentration column** is used for all samples in which asbestos is observed either under stereomicroscopy or in PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be similar to or greater than the 0.2 % reference material but less than the 1% reference material (tan and/or brown, whichever is most similar in appearance to the test material). These samples are assigned to **Bin B2**.

**A numeric value (1, 2, 3, etc) in the Concentration column without an entry in the Qualifier column** is used for all samples in which asbestos is observed either under stereomicroscopy or in PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be similar to or greater than the 1 % reference material (tan and/or brown, whichever is most similar in appearance to the test material). These samples are assigned to **Bin C**.

Note that because these reference materials are based on Libby amphibole, they are not appropriate for estimating the mass percent of other types of asbestos (chrysotile, other types of asbestos). Therefore, if any asbestos types besides Libby amphibole are observed, the reported values for those samples should be in units of area percent.

### **5.3 Point Counting Approach**

#### **5.3.1 Counting Procedure**

Any analysis in which evaluation by point counting is requested will be performed in general accordance with the descriptions provided in EPA/600/R-93/116 and CARB Method 435. The total number of particles to be counted (generally 400 or 1000) will be specified in the Chain of Custody request.

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Take eight sub-samples of the soil sample and mount each separately with the appropriate refractive index liquid. The preparations should not be heavily loaded. Each sample should be uniformly dispersed to avoid overlapping particles and allow 25-50% empty area within the fields of view.

An ocular reticule (point array) or cross-hair is used to visually superimpose points on the microscope field of view. Count 1/8 of the total points required on each of the 8 slides (e.g., 50 non-empty points per slide for a 400 point count and 125 non-empty points per slide for a 1000 point count). For each non-empty point counted, assign the particle that is present at the point into one of four bins:

- Not asbestos
- Libby asbestos (LA)
- Other asbestos (OA)
- Chrysotile asbestos (C)

In order for a particle to be counted as asbestos, the aspect ratio must be  $\geq 3:1$ .

After the required total number of non-empty points have been counted, record the total number of points in the LA, OA and C bins on the point counting data sheet.

#### *5.3.2 Estimation of Mass Percent*

Like visual estimation, the output of the point counting approach is an estimate of area fraction, not mass fraction. For this site, point-count estimates of area fraction for Libby amphibole particles will be converted into estimates of mass fraction using a standard curve approach.

The standard curve will be prepared using a series of site-specific reference materials containing 0%, 0.2%, 0.5%, 1%, or 2% Libby amphibole. The area fraction of each reference material will be estimated by the point counting approach in quadruplicate. The standard curve will be prepared by plotting the mean area fraction determined by point counting versus the mass percent in the reference material. The mass fraction of a field sample will be determined by measuring the area fraction of the field sample and locating the mass fraction that corresponds to that area fraction on the standard curve.

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Because the standard curve is based on Libby amphibole, it is not appropriate to utilize this standard curve for other types of asbestos. Therefore, if any asbestos types besides Libby amphibole are observed, the reported values for those samples should be in units of area percent.

## **6.0 APPARATUS AND MATERIALS**

Polarized light microscope, with lens and filters  
Stereomicroscope (approximately 10-45x)  
Petri dish for stereomicroscopic sample examination  
Spatula and forceps  
Glass slides and cover slips  
Refractive Index (RI) oils  
Reference Materials  
    Tan soil, 0.2% LA by mass  
    Tan soil, 1.0% LA by mass  
    Brown Soil, 0.2% LA by mass  
    Brown soil, 1.0% LA by mass  
Laboratory log book  
Data recording sheet (Attachment 1)

## **7.0 QUALITY ASSURANCE/QUALITY CONTROL**

### **7.1 Precision and Accuracy**

PLM by visual estimation and point counting are both semi-quantitative methods. For the purposes of this project, the accuracy and precision of the method are evaluated by measuring the frequency with which samples are assigned to the correct "bins". Data on precision and accuracy of bin assignment will be collected in the future and used to establish performance criteria for this project.

### **7.2 Method Proficiency**

At present, sufficient data are not available to establish a quantitative procedure for method proficiency demonstration. As results become available, a procedure will be established and applied, based on the analysis of a set of blind Performance Evaluation materials and assessing



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the frequency of correct bin assignments. If the assignments reported by a laboratory are within acceptance criteria bounds (see Section 7.1), then that laboratory will be deemed proficient. If not, remedial actions must be taken to address the errors before work may begin by that laboratory.

## **8.0 RECORDS**

### **8.1 PLM Data Forms**

Analysts will record analytical results using the electronic data sheets developed for the Libby project, as presented in Attachment 1. Note that there are two different electronic forms; one is for use in visual area estimation, and the other is for use in point counting. Once completed and checked, these spreadsheets are submitted to EPA for upload into the database. The laboratory should retain all original records for use in resolving any questions until otherwise instructed by EPA.

### **8.2 Instrument Maintenance Logbook**

An individual instrument maintenance logbook should be kept for each piece of equipment in use at the laboratory. All maintenance activities must be recorded in the appropriate logbook.

### **8.3 Data Storage and Archival**

**Electronic Data.** Each day of data acquisition, all electronic files will be saved onto two separate media. For example, the data may be saved onto a computer hard drive, but must also be backed up onto a type of portable media such as CD-ROM, floppy disc, or tape. Portable media will be maintained in a single location with limited access.

**Hardcopy Data.** All data sheets and micrographs must be stored in a secured location with limited access (e.g., locking file cabinet) when not in use.

Copies (hardcopy and electronic) of the raw analytical data will be submitted to USEPA for archival.

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## 9.0 REFERENCES

CARB 435. California Environmental Protection Agency Air Resources Board, Method 435, Determination of Asbestos Content in Serpentine Aggregate. June 6, 1991.

EPA. 1993. Method for the Determination of Asbestos in Bulk Building Materials. United States Environmental Protection Agency, Office of Research and Development. EPA/600/R-93/116. July 1993.

EPA. 2003. Technical Memo 8. Procedure for Combining Mass Fraction Estimates for Coarse and Fine Fractions of Soil. Prepared by US EPA Region 8 with technical assistance from Syracuse Research Corporation.

NIOSH. 1994. Asbestos (Bulk) by PLM. NIOSH Manual of Analytical Methods, Fourth Edition. National Institute of Occupational Safety and Health. August 15, 1994.

Wylie AG and Verkouteren JR. 2000. Amphibole Asbestos from Libby, Montana: Aspects of Nomenclature. *American Mineralogist* 85:1540-1542.

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**ATTACHMENT 1**

**PLM DATA RECORDING SHEETS**

**PLM (VE and PC) Data Sheet and EDD v4.xls**

**ASBESTOS (bulk) by PLM****9002**

various

MW: various

CAS: 1332-21-4

RTECS: C16475000

**METHOD:** 9002, Issue 2**EVALUATION:** PARTIAL**Issue 1:** 15 May 1989**Issue 2:** 15 August 1994**EPA Standard (Bulk):** 1%**PROPERTIES:** solid, fibrous, crystalline, anisotropic

**SYNONYMS [CAS #]:** actinolite [77536-66-4], or ferroactinolite [15669-07-5]; amosite [12172-73-5]; anthophyllite [77536-67-5]; chrysotile [12001-29-5]; serpentine [18786-24-8]; crocidolite [12001-28-4]; tremolite [77536-68-6]; amphibole.

SAMPLING		MEASUREMENT	
<b>BULK SAMPLE:</b>	1 to 10 grams	<b>TECHNIQUE:</b>	MICROSCOPY, STEREO AND POLARIZED LIGHT, WITH DISPERSION STAINING
<b>SHIPMENT:</b>	seal securely to prevent escape of asbestos	<b>ANALYTE:</b>	actinolite asbestos, amosite, anthophyllite asbestos, chrysotile, crocidolite, tremolite asbestos
<b>SAMPLE STABILITY:</b>	stable	<b>EQUIPMENT:</b>	microscope, polarized light; 100-400X dispersion staining objective, stereo microscope: 10-45X
<b>BLANKS:</b>	none required	<b>RANGE:</b>	1% to 100% asbestos
<b>ACCURACY</b>		<b>ESTIMATED LOD:</b>	<1% asbestos [1]
<b>RANGE STUDIED:</b>	<1% to 100% asbestos	<b>PRECISION:</b>	not determined
<b>BIAS:</b>	not determined		
<b>PRECISION:</b>	not determined		
<b>ACCURACY:</b>	not determined		

**APPLICABILITY:** this method is useful for the qualitative identification of asbestos and the semi-quantitative determination of asbestos content of bulk samples. The method measures percent asbestos as perceived by the analyst in comparison to standard area projections, photos, and drawings, or trained experience. The method is not applicable to samples containing large amounts of fine fibers below the resolution of the light microscope

**INTERFERENCES:** Other fibers with optical properties similar to the asbestos minerals may give positive interferences. Optical properties of asbestos may be obscured by coating on the fibers. Fibers finer than the resolving power of the microscope (ca. 0.3  $\mu\text{m}$ ) will not be detected. Heat and acid treatment may alter the index of refraction of asbestos and change its color.

**OTHER METHODS:** This method (originally designated as method 7403) is designed for use with NIOSH Methods 7400 (phase contrast microscopy) and 7402 (electron microscopy/EDS). The method is similar to the EPA bulk asbestos method [1].

#### REAGENTS:

1. Refractive index (RI) liquids for Dispersion Staining: high-dispersion (HD) series, 1.550, 1.605, 1.620.
2. Refractive index liquids: 1.670, 1.680, and 1.700.
3. Asbestos reference samples such as SRM #1866, available from the National Institute of Standards and Technology.\*
4. Distilled Water (optional).
5. Concentrated HCl: ACS reagent grade.

\* See SPECIAL PRECAUTIONS

#### EQUIPMENT:

1. Sample containers: screw-top plastic vials of 10- to 50-mL capacity.
2. Microscope, polarized light, with polarizer, analyzer, port for retardation plate, 360° graduated rotating stage, substage condenser with iris, lamp, lamp iris, and:
  - a. Objective lenses: 10X, 20X, and 40X or near equivalent.
  - b. Ocular lense: 10X minimum.
  - c. Eyepiece reticle: crosshair.
  - d. Dispersion staining objective lens or equivalent.
  - e. Compensator plate: ca. 550 nm± 20 nm, retardation: "first order red" compensator.
3. Microscope slides: 75 mm x 25 mm.
4. Cover slips.
5. Ventilated hood or negative-pressure glove box.
6. Mortar and pestle: agate or porcelain.
7. Stereomicroscope, ca. 10 to 45X.
8. Light source: incandescent or fluorescent.
9. Tweezers, dissecting needles, spatulas, probes, and scalpels.
10. Glassine paper or clean glass plate.
11. Low-speed hand drill with coarse burr bit (optional).

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**SPECIAL PRECAUTIONS:** Asbestos, a human carcinogen, should be handled only in an exhaust hood (equipped with a HEPA filter) [2]. Precautions should be taken when collecting unknown samples, which may be asbestos, to preclude exposure to the person collecting the sample and minimize the disruption to the parent material [3]. Disposal of asbestos-containing materials should follow EPA Guidelines [4].

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#### SAMPLING:

1. Place 1 to 10 g of the material to be analyzed in a sample container.

NOTE: For large samples (i.e., whole ceiling tiles) that are fairly homogenous, a representative small portion should be submitted for analysis. Sample size should be adjusted to ensure that it is representative of the parent material.
2. Make sure that sample containers are taped so they will not open in transit.
3. Ship the samples in a rigid container with sufficient packing material to prevent damage or sample loss.

#### SAMPLE PREPARATION:

4. Visually examine samples in the container and with a low-magnification stereomicroscope in a hood. (If necessary, a sample may be carefully removed from the container and placed on glassine transfer paper or clean glass plate for examination). Break off a portion of the sample and examine the edges for emergent fibers. Note the homogeneity of the sample. Some hard tiles can be broken, and the edges examined for emergent fibers. If fibers are found, make an estimate of the amount and type of fibers present, confirm fiber type (step 14) and quantify (step 15).
5. In a hood, open sample container and with tweezers remove small, representative portions of the sample.
  1. If there are obvious separable layers, sample and analyze each layer separately.

- b. If the sample appears to be slightly inhomogeneous, mix it in the sample container with tweezers or a spatula before taking the portion of analysis. Alternatively, take small representative portions of each type of material and place on a glass slide.
- c. On hard tiles that may have thin, inseparable layers, use a scalpel to cut through all the layers for a representative sample. Then cut it into smaller pieces after placing RI liquid on it before trying to reduce the thickness. Alternatively, use a low-speed hand drill equipped with a burr bit to remove material from hard tiles. Avoid excessive heating of the sample which may alter the optical properties of the material.

NOTE: This type of sample often requires ashing or other specialized preparation, and may require transmission electron microscopy for detection of the short asbestos fibers which are characteristic of floor tiles.

- d. If the sample has large, hard particles, grind it in a mortar. Do not grind so fine that fiber characteristics are destroyed.
- e. If necessary, treat a portion of the sample in a hood with an appropriate solvent to remove binders, tars, and other interfering materials which may be present in the sample. Make corrections for the non-asbestos material removed by this process.

NOTE: Other methods of sample preparation such as acid washing and sodium metaphosphate treatment and ashing may be necessary, especially to detect low concentrations of asbestos. If needed, use as described in Reference [1].

- 6. After placing a few drops of RI liquid on the slide, put a small portion of sample in the liquid. Tease apart with a needle or smash small clumps with the flat end of a spatula or probe, producing a uniform thickness or particles so that better estimates of projected area percentages can be made. Mix the fibers and particles on the slide so that they are as homogeneous as possible.

NOTE: An even dispersion of sample should cover the entire area under the cover slip. Some practice will be necessary to judge the right amount of material to place on the slide. Too little sample may not give sufficient information and too much sample cannot be easily analyzed.

#### **CALIBRATION AND QUALITY CONTROL:**

- 7. Check for contamination each day of operation. Wipe microscope slides and cover slips with lens paper before using. Check refractive index liquids. Record results in a separate logbook.
- 8. Verify the refractive indices of the refractive index liquids used once per week of operation. Record these checks in a separate logbook.
- 9. Follow the manufacturer's instructions for illumination, condenser alignment and other microscope adjustments. Perform these adjustments prior to each sample set.
- 10. Determine percent of each identified asbestos species by comparison to standard projections (Figure 1) [1]. If no fibers are detected in a homogeneous sample, examine at least two additional preparations before concluding that no asbestos is present.
- 11. If it appears that the preparation technique might not be able to produce a homogeneous or representative sample on the slide, prepare a duplicate slide and average the results. Occasionally, when the duplicate results vary greatly, it will be necessary to prepare additional replicate slides and average all the replicate results. Prepare duplicate slides of at least 10% of the samples analyzed. Average the results for reporting.
- 12. Analyze about 5% blind samples of known asbestos content.
- 13. Laboratories performing this analytical method should participate in the National Voluntary Laboratory Accreditation Program [5] or a similar interlaboratory quality control program. Each analyst should have complete formal training in polarized light microscopy and its application to crystalline materials. In lieu of formal training, laboratory training in asbestos bulk analysis under the direction of a trained asbestos bulk analyst may be substituted. Owing to the subjective nature of the method, frequent practice is essential in order to remain proficient in estimating projected area percentages.

#### **QUALITATIVE ASSESSMENT:**

- 14. Scan the slide to identify any asbestos minerals using the optical properties of morphology,

refractive indices, color, pleochroism, birefringence, extinction characteristics, sign of elongation, and dispersion staining characteristics.

NOTE: Identification of asbestos using polarized light microscopy is unlike most other analytical methods. The quality of the results is dependent on the skill and judgment of the analyst. This method does not lend itself easily to a step-wise approach. Various procedures devised by different analysts may yield equivalent results. The following step-wise procedure repeatedly utilizes the sample preparation procedure previously outlined.

- a. Prepare a slide using 1.550 HD RI liquid. Adjust the polarizing filter such that the polars are partially crossed, with ca. 15° offset. Scan the preparation, examining the morphology for the presence of fibers. If no fibers are found, scan the additional preparations. If no fibers are found in any of the preparations, report that the sample does not contain asbestos, and stop the analysis at this point.
- b. If fibers are found, adjust the polarizing filter such that the polars are fully crossed. If all of the fibers are isotropic (disappear at all angles of rotation) then those fibers are not asbestos. Fibrous glass and mineral wool, which are common components of suspect samples, are isotropic. If only isotropic fibers are found in the additional preparations, report no asbestos fibers detected, and stop the analysis.
- c. If anisotropic fibers are found, rotate the stage to determine the angle of extinction. Except for tremolite-actinolite asbestos which has oblique extinction at 10-20°, the other forms of asbestos exhibit parallel extinction (Table 1). Tremolite may show both parallel and oblique extinction.
- d. Insert the first order red compensator plate in the microscope and determine the sign of elongation. All forms of asbestos have a positive sign of elongation except for crocidolite. If the sign of elongation observed is negative, go to step "g."

NOTE: To determine the direction of the sign of elongation on a particular microscope configuration, examine a known chrysotile sample and note the direction (NE-SW or NW-SE) of the blue coloration. Chrysotile has a positive sign of elongation.

- e. Remove the first-order red compensator and uncross the polarizer. Examine under plane polarized light for blue and gold-brown Becke colors at the fiber-oil interface (i.e., index of refraction match). Becke colors are not always evident. Examine fiber morphology for twisted, wavy bundles of fibers which are characteristic of chrysotile. Twisted, ribbon-like morphology with cellular internal features may indicate cellulose fibers. It may be necessary to cross the polars partially in order to see the fibers if the index of refraction is an exact match at 1.550. If the fibers appear to have higher index of refraction, go to step "h," otherwise continue.
- f. Identification of chrysotile. Insert the dispersion staining objective. Observation of dispersion staining colors of blue and blue-magenta confirms chrysotile. Cellulose, which is a common interfering fiber at the 1.550 index of refraction, will not exhibit these dispersion staining colors. If chrysotile is found, go to step 15 for quantitative estimation.
- g. Identification of crocidolite. Prepare a slide in 1.700 RI liquid. Examine under plane-polarized light (uncrossed polars); check for morphology of crocidolite. Fibers will be straight, with rigid appearance, and may appear blue or purple-blue. Crocidolite is pleochroic, i.e., it will appear to change its color (blue or gray) as it is rotated through plane polarized light. Insert the dispersion staining objective. The central stop dispersion staining color are red magenta and blue magenta, however, these colors are sometimes difficult to impossible to see because of the opacity of the dark blue fibers. If observations above indicate crocidolite, go to step 15 for quantitative estimation.
- h. Identification of amosite. Prepare a slide in 1.680 RI liquid. Observed the fiber morphology for amosite characteristics: straight fibers and fiber bundles with broom-like or splayed ends. If the morphology matches amosite, examine the fibers using the dispersion staining objective. Blue and pale blue colors indicate the cummingtonite form of amosite, and gold and blue colors indicate the grunerite form of amosite. If amosite is confirmed by this test, go to step 15 for quantitative estimation, otherwise continue.
- i. Identification of anthophyllite-tremolite-actinolite. Prepare a slide in 1.605 HD RI liquid. Examine morphology for comparison to anthophyllite-tremolite-actinolite asbestos. The refractive indices for these forms of asbestos vary naturally within the species. Anthophyllite can be distinguished from actinolite and tremolite by its nearly parallel extinction. Actinolite has a light to dark green color under plane-polarized light and exhibits some pleochroism. For all

three, fibers will be straight, single fibers possibly with some larger composite fibers. Cleavage fragments may also be present. Examine using the central stop dispersion staining objective. Anthophyllite will exhibit central stop colors of blue and gold/gold-magenta; tremolite will exhibit pale blue and yellow; and actinolite will exhibit magenta and golden-yellow colors.

NOTE: In this refractive index range, wollastonite is a common interfering mineral with similar morphology including the presence of cleavage fragments. It has both positive and negative sign of elongation, parallel extinction, and central stop dispersion staining colors of pale yellow and pale yellow to magenta. If further confirmation of wollastonite versus anthophyllite is needed, go to step "j". If any of the above forms of asbestos were confirmed above, go to step 15 for quantitative estimation. If none of the tests above confirmed asbestos fibers, examine the additional preparations and if the same result occurs, report the absence of asbestos in this sample.

- j. Wash a small portion of the sample in a drop of concentrated hydrochloric acid on a slide. Place the slide, with cover slip in place, on a warm hot plate until dry. By capillary action, place 1.620 RI liquid under the cover clip and examine the slide. Wollastonite fibers will have a "cross-hatched" appearance across the length of the fibers and will not show central stop dispersion colors. Anthophyllite and tremolite will still show their original dispersion colors.

NOTE: There are alternative analysis procedures to the step-wise approach outlined above which will yield equivalent results. Some of these alternatives are:

- i. Perform the initial scan for the presence of asbestos using crossed polars as well as the first-order red compensator. This allows for simultaneous viewing of birefringent and amorphous materials as well as determine their sign of elongation. Some fibers which are covered with mortar may best be observed using this configuration.
- ii. Some analysts prefer to mount their first preparation in a RI liquid different than any asbestos materials and conduct their initial examination under plane-polarized light.
- iii. If alternative RI liquids are used from those specified, dispersion staining colors observed will also change. Refer to an appropriate reference for the specific colors associated with asbestos in the RI liquids actually used.

#### QUANTITATIVE ASSESSMENT:

15. Estimate the content of the asbestos type present in the sample using the 1.550 RI preparation. Express the estimate as an area percent of all material present, taking into account the loading and distribution of all sample material on the slide. Use Figure 1 as an aid in arriving at your estimate. If additional unidentified fibers are present in the sample, continue with the qualitative measurement (step 14).

NOTE: Point-counting techniques to determine percentages of the asbestos minerals are not generally recommended. The point-counting method only produces accurate quantitative data when the material on the slide is homogeneous and has a uniform thickness, which is difficult to obtain [6]. The point-counting technique is, recommended by the EPA to determine the amount of asbestos in bulk [1]; however, in the more recent Asbestos Hazard Emergency Response Act (AHERA) regulations, asbestos quantification may be performed by a point-counting or equivalent estimation method [7].

16. Make a quantitative estimate of the asbestos content of the sample from the appropriate combination of the estimates from both the gross and microscopic examinations. If asbestos fibers are identified, report the material as "asbestos-containing". Asbestos content should be reported as a range of percent content. The range reported should be indicative of the analyst's precision in estimating asbestos content. For greater quantities use Figure 1 in arriving at your estimate.

#### EVALUATION OF METHOD:

The method is compiled from standard techniques used in mineralogy [8-13], and from standard laboratory procedures for bulk asbestos analysis which have been utilized for several years. These



techniques have been successfully applied to the analysis of EPA Bulk Sample Analysis Quality Assurance Program samples since 1982 [1,5]. However, no formal evaluation of this method, as written, has been performed.

#### REFERENCES:

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#### METHOD WRITTEN BY:

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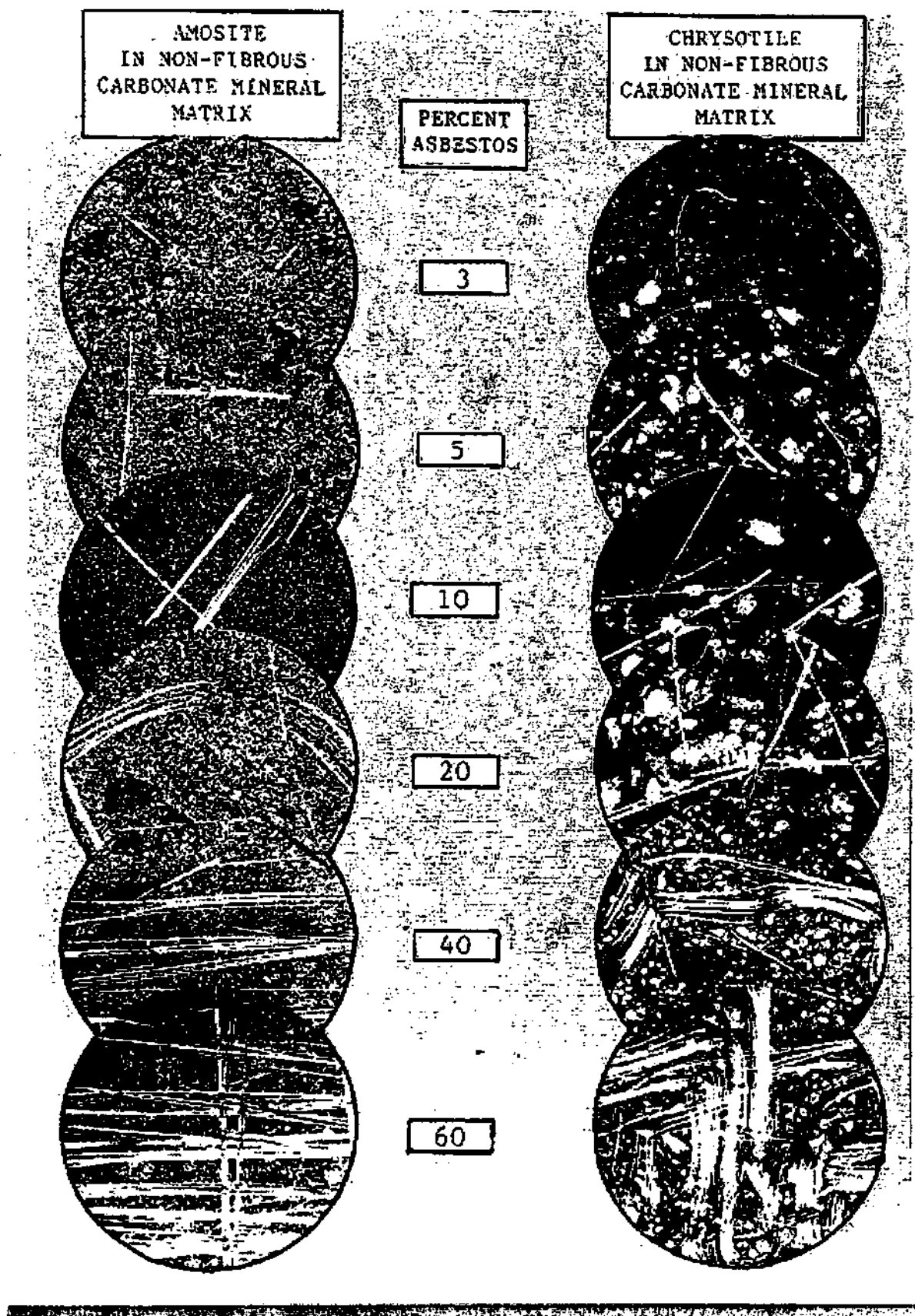


Figure 1. Percent estimate comparator

Table 1. Optical Properties of Asbestos Fibers				
Mineral	Morphology and Color	Refractive Index (Approximate Values)		Birefringence
		$\perp$ to Elongation	$\parallel$ to Elongation	
Chrysotile	Wavy fibers with kinks. Splayed ends on larger bundles. Colorless to light brown upon being heated. Nonpleochroic. Aspect ratio typically >10:1.	1.54	1.55	0.002 - 0.014
Cummingtonite- Grunerite (Amosite)	Straight fibers and fiber bundles. Bundle ends appear broom-like or splayed. Colorless to brown upon heating. May be weakly pleochroic. Aspect ratio typically >10:1.	1.67	1.70	0.02 - 0.03
Crocidolite (Riebeckite)	Straight fibers and fiber bundles. Longer fibers show curvature. Splayed ends on bundles. Characteristic blue color. Pleochroic. Aspect ratio typically >10:1.	1.71	1.70	0.014 - 0.016 Interference colors may be masked by blue color.
Anthophyllite	Straight fibers and fiber bundles. Cleavage fragments may be present. Colorless to light brown. Nonpleochroic to weakly pleochroic. Aspect ratio generally <10:1.	1.61	1.63	0.019 - 0.024
Tremolite- Actinolite	Straight and curved fibers. Cleavage fragments common. Large fiber bundles show splayed ends. Tremolite is colorless. Actinolite is green and weakly to moderately pleochroic. Aspect ratio generally <10:1.	1.60 - 1.62 (tremolite)  1.62 - 1.67 (actinolite)	1.62 - 1.64 (tremolite)  1.64 - 1.68 (actinolite)	0.02 - 0.03

Table 1. Optical Properties of Asbestos Fibers (Continued)					
Mineral	Extinction	Sign of Elongation	Central Stop Dispersion Staining Colors		
			RI Liquid	$\perp$ to Vibration	$\parallel$ to Vibration
Chrysotile	Parallel to fiber length	+ (length slow)	1.550 <sup>HD</sup>	Blue	Blue-magenta
Cummingtonite- Grunerite (Amosite)	Parallel to fiber length	+ (length slow)	1.670 Fibers subjected to high temperatures will not dispersion-stain.	Red magenta to blue	Yellow
Cummingtonite Grunerite			1.680 1.680	pale blue blue	blue gold
Crocidolite (Riebeckite)	Parallel to fiber length	- (length fast)	1.700 1.680	Red magenta yellow	Blue-magenta pale yellow
Anthophyllite	Parallel to fiber length	+ (length slow)	1.605 <sup>HD</sup> 1.620 <sup>HD</sup>	Blue Blue-green	Gold to gold-magenta Golden-yellow
Tremolite- Actinolite	Oblique - 10 to 20° for fragments. Some composite fibers show $\parallel$ extinction.	+ (length slow)	1.605 <sup>HD</sup>	Pale blue (tremolite) Yellow (actinolite)	Yellow (tremolite) Pale yellow (actinolite)

HD = high-dispersion RI liquid series.

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DOR  
DecDate: November 12, 2002SOP No. SRC-LIBBY-01 (Revision 0)

Title: QUALITATIVE ESTIMATION OF ASBESTOS IN COARSE SOIL BY VISUAL EXAMINATION USING STEREOMICROSCOPY AND POLARIZED LIGHT MICROSCOPY

Author Sally M. L. Gibson

Syracuse Research Corporation

**SYNOPSIS:** A standardized method is described for the examination of the coarse fraction (>1/4") of soil samples using stereomicroscopy and polarized light microscopy (PLM) to identify, segregate, and estimate the mass percent of asbestos in the sample matrix.

Received by QA Unit:

**APPROVALS:****TEAM MEMBER****SIGNATURE/TITLE****DATE**EPA Region 8Jaeey Goldsade11/12/02Syracuse Research Corp.WJ Brattens11/12/02

Revision	Date	Reason for Revision
0	11/12/02	--

**TECHNICAL STANDARD OPERATING PROCEDURE**  
**SRC-LIBBY-01**

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## **1.0 PURPOSE**

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized screening method for the visual examination of the coarse fraction of previously sieved soil samples for evidence of asbestos mineral content using stereomicroscopy with confirmation of asbestos content by polarized light microscopy (PLM.) This SOP incorporates salient components of EPA Test Method 600/R-93/116 *Method for Determination of Asbestos in Bulk Building Materials* and National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

This procedure will be used by employees of contractors/subcontractors supporting USEPA Region 8 projects and tasks for the Libby, Montana site. Deviations from the procedure outlined in this document must be approved by the USEPA Region 8 Remedial Project Manager or Regional Chemist prior to initiation of sample analysis.

## **2.0 PREREQUISITE TRAINING**

Visual examination will be performed according to this SOP by a laboratory accredited by the National Voluntary Laboratory Accreditation Program (NVLAP) and by analysts proficient either by education or experience in asbestos mineral identification by stereomicroscopy and PLM. Analyst familiarity with the procedural applications prescribed in EPA Test Method 600/R-93/116 and NIOSH Method 9002 is required.

Training as described in the Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4, (CSS SQAPP [CDM 2002]) will be provided to laboratory personnel or laboratories with less than 1 year project-specific experience by "mentors" from either Reservoir Environmental Services, Inc. or EMSL.

## **3.0 RESPONSIBILITIES**

The CDM Laboratory Coordinator (LC) is responsible for overseeing the activities of the CDM Soil Preparation Laboratory and subcontracted laboratories performing sample analysis for the Libby, Montana project. The LC is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the CSS SQAPP. It is the responsibility of the LC to communicate with the project personnel and subcontracted laboratory regarding specific analysis objectives and anticipated situations that require any deviation from the CSS SQAPP SOPs. In addition, it is the responsibility of the LC to communicate the need for any deviations from this SOP with the CDM Project Manager, USEPA Region 8 personnel (Remedial Project Manager or Regional Chemist.)

## TECHNICAL STANDARD OPERATING PROCEDURE

### SRC-LIBBY-01

Subcontracted laboratory analysts performing the visual examination are responsible for adhering to the applicable tasks outlined in this SOP and substantiating components of the reference procedures (EPA 1993; NIOSH 1994) with the modifications contained herein.

#### 4.0 EQUIPMENT

- Analytical balance - accurate to 0.1 g, range of 0.1 g to 1000 g
- Traceable standards - major asbestos types
- Microscope - binocular stereomicroscope, 5-60X approximate magnification
- Microscope - polarized light, binocular or monocular with a cross hair reticle (or functional equivalent) and magnification of at least 8X
  - 10X, 20X, and 40X objectives
  - 360 degree rotatable stage
  - substage condenser with iris diaphragm
  - polarizer and analyzer which can be placed at 90 degrees to one another and calibrated relative to the cross-line reticle in the ocular
  - port for wave plates and compensators
  - wave retardation plate (Red I Compensator) with ~550 nanometer retardation and known slow and fast vibration directions
- Light Sources - incandescent or fluorescent
- Tweezers, dissecting needles, scalpels, probes, razor knives, etc. - standard sample manipulation instruments/tools
- Microscope slides and cover slips
- Refractive index liquids
- Pre-tared glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. - laboratory sample containers
- HEPA-filtered or Class 1 biohazard hood negative pressure
- Three-ring binder book - binders will contain Microscopic Examination Logbook Sheets (Attachment 1)

TECHNICAL STANDARD OPERATING PROCEDURE  
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## 5.0 METHOD

Soils from the Libby, Montana site will be dried, sieved, and prepared according to the most recent revision of SOP ISSI-LIBBY-01, Soil Sample Preparation. The coarse fraction of the soil sample is defined as that portion of the sample which does not pass through a 1/4" sieve. The coarse fraction will be weighed, placed in a zip-top plastic bag, and labeled as described in Camp, Dresser and McKee (CDM) SOP 1-3 (with project-specific modifications). The samples will be packaged and shipped by the soil preparation laboratory as described in CDM SOP 2-1 (with project-specific modifications) and transferred to the laboratory via chain-of-custody procedures described in CDM SOP 1-2 (with project-specific modifications).

The following sections describe the stereomicroscopic and PLM examination. Materials tentatively characterized as asbestos by stereomicroscopy will be isolated and subjected to confirmation by PLM. The mass % of amphibole asbestos in the coarse soil fraction will be calculated using the mass of amphibole asbestos positively identified by PLM and the original sample weight. Figure 1 provides an overview of the process.

### 5.1 Stereomicroscopic Examination

The laboratory will receive the coarse fraction soil samples from the CDM Soil Preparation Laboratory. The entire sample will be weighed and placed in an appropriate, pre-tared container. The weights will be recorded, along with the sample identification, on the Microscope Examination Logbook Sheet. The sample will be subject to stereomicroscopic examination to characterize the coarse fraction as either as depicted Figure 1. As the examination proceeds, the stereomicroscopist will transfer any tentatively identified asbestos into a pre-tared sample container or sample boat. The stereomicroscopic examination to identify and segregate asbestos includes:

- using multiple fields of view over the entire sample
- probing the sample by turning pieces over and breaking clumps where possible
- manipulating the sample using appropriate instruments/tools
- observing homogeneity, texture, friability, color and extent of any observed asbestos in the sample(s)

As the sample is examined, the analyst will continue segregation of the sample until the entire coarse soil fraction has been characterized as either "non-asbestos" or "tentatively identified asbestos". The stereomicroscopist will separate to the extent possible distinctly different materials (i.e., layered) and perform appropriate manipulation techniques to allow the asbestos materials to be separated for easier identification. Any material determined to be non-asbestos will be set aside with the original non-asbestos portion of the sample. The remaining tentatively identified amphibole asbestos fraction will be examined by PLM. The stereomicroscopist will initial and date the Microscopy Examination Logbook Sheet.



TECHNICAL STANDARD OPERATING PROCEDURE  
SRC-LIBBY-01

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## 5.2 PLM

The coarse material tentatively identified as amphibole asbestos by stereomicroscopic examination will be subject to confirmation examination by PLM. The PLM examination will be used to confirm that the particles tentatively classified as asbestos are actually asbestos.

The iterative process of preparation, analysis, and identification of samples examined by PLM is detailed in EPA (1993) and NIOSH (1994). In general, randomly selected subsamples of the tentatively identified asbestos will be mounted in appropriate refractive index liquids allowing the fibers to be distinguishable from non-asbestos components. The total weight of positively identified asbestos will be determined and recorded on the Microscopic Examination Logbook Sheet, along with the analyst's initials, and the date of the examination. Any particles that are confirmed by PLM as non-asbestos will be removed before weighing, and returned to the original non-LA asbestos portion of the sample. To the extent possible, all asbestos structures will be classified either as amphibole or chrysotile, and these will be weighted separately. This determination allows for the mass % to be reflected in terms of % amphibole asbestos and/or % chrysotile asbestos.

## 6.0 QUALITY ASSURANCE

Laboratories performing the examination must be accredited by NVLAP. "Calibration" should be verifiable for each microscopist in terms of project-specific training and the successful analysis of materials of known asbestos content (NVLAP test samples, in-house standards) similar to those anticipated to be observed in Libby, Montana soils. Additionally, references such as photographs of the asbestos minerals illustrating distinguishing properties should be available benchside during characterization.

Quality control samples as described in ISSI-LIBBY-01 (i.e., preparation duplicates) will not be submitted for the coarse materials samples. The entire coarse fraction will be subject to examination.

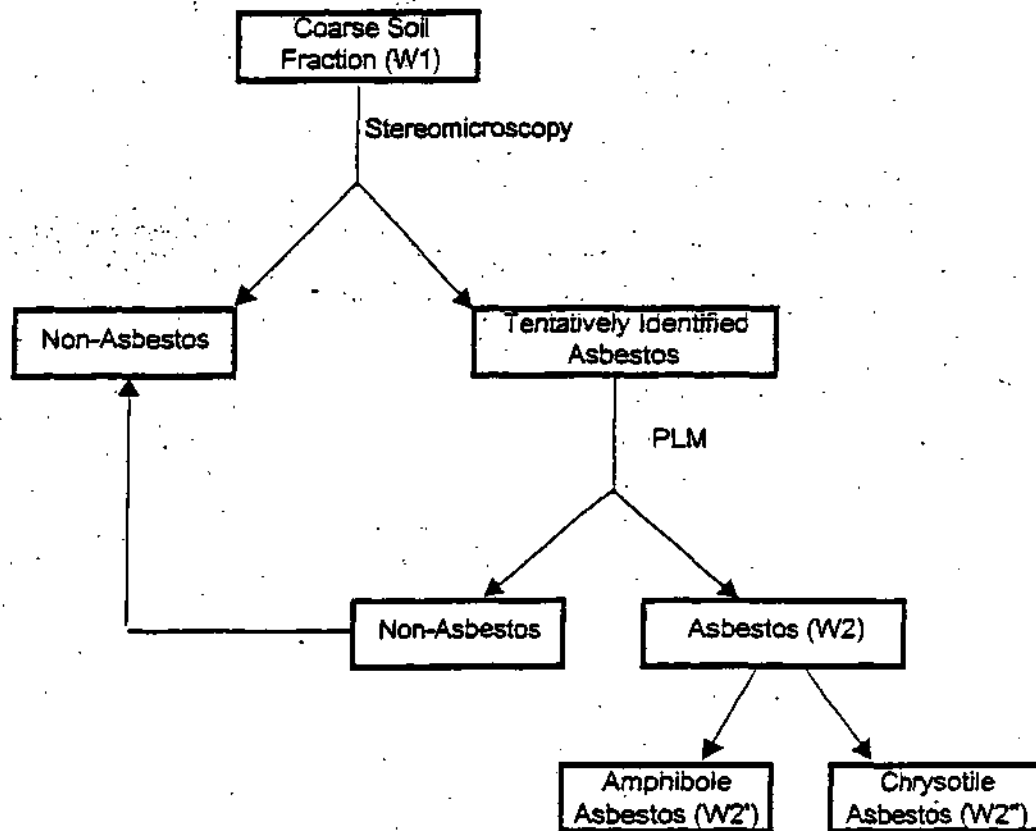
## 7.0 REFERENCES

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NIOSH 1994. National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

USEPA 1993. *Method for Determination of Asbestos in Bulk Building Materials*. 600/R-93/116.

Figure 1. Overview of Sample Examination Process



W1 = Original coarse soil fraction weight (g)

W2 = Mass of asbestos (g)

W2' = If present in measurable quantities, mass (g) of amphibole

W2'' = If present in measurable quantities, mass (g) of chrysotile

TECHNICAL STANDARD OPERATING PROCEDURE  
SRC-LIBBY-01

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ATTACHMENT 1  
MICROSCOPIC EXAMINATION LOGBOOK SHEET

## Microscopic Examination Logbook Sheet

[illegible]

Number codes = additional observation enumerated in the Notes section

**Notes:**

### Calculations

Mass of sample =  $(W_2 - W_1) = W_3$

$$\text{Mass of 1 N.A} = (VW3 - VW4) = VW8$$
$$\% \text{ I.A.} = (W7 / W3) \times 100$$
$$\% \text{ chrysotile} = (W_0 / W_3) \times 100$$

**Electronic Code of Federal Regulations**

**e-CFR**

THIS DATA CURRENT AS OF THE FEDERAL REGISTER DATED JULY 12, 2002

**40 CFR - CHAPTER I - PART 763**

**View Part**

**Appendix A to Subpart E of Part 763 -- Interim Transmission Electron Microscopy Analytical Methods -- Mandatory and Nonmandatory -- and Mandatory Section to Determine Completion of Response Actions**

**I. Introduction**

The following appendix contains three units. The first unit is the mandatory transmission electron microscopy (TEM) method which all laboratories must follow; it is the minimum requirement for analysis of air samples for asbestos by TEM. The mandatory method contains the essential elements of the TEM method. The second unit contains the complete non-mandatory method. The non-mandatory method supplements the mandatory method by including additional steps to improve the analysis. EPA recommends that the non-mandatory method be employed for analyzing air filters; however, the laboratory may choose to employ the mandatory method. The non-mandatory method contains the same minimum requirements as are outlined in the mandatory method. Hence, laboratories may choose either of the two methods for analyzing air samples by TEM.

The final unit of this Appendix A to subpart E defines the steps which must be taken to determine completion of response actions. This unit is mandatory.

**II. Mandatory Transmission Electron Microscopy Method**

**A. Definitions of Terms**

1. *Analytical sensitivity* -- Airborne asbestos concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 structures/cm<sup>3</sup>.
2. *Asbestiform* -- A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.
3. *Aspect ratio* -- A ratio of the length to the width of a particle. Minimum aspect ratio as defined by

this method is equal to or greater than 5:1.

4. *Bundle* -- A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

5. *Clean area* -- A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200-mesh grid openings) and a maximum of 53 structures/mm<sup>2</sup> for any single preparation for that same area.

6. *Cluster* -- A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

7. *ED* -- Electron diffraction.

8. *EDXA* -- Energy dispersive X-ray analysis.

9. *Fiber* -- A structure greater than or equal to 0.5  $\mu$ m in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.

10. *Grid* -- An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.

11. *Intersection* -- Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.

12. *Laboratory sample coordinator* -- That person responsible for the conduct of sample handling and the certification of the testing procedures.

13. *Filter background level* -- The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on a blank (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm<sup>2</sup>.

14. *Matrix* -- Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

15. *NSD* -- No structure detected.

16. *Operator* -- A person responsible for the TEM instrumental analysis of the sample.

17. *PCM* -- Phase contrast microscopy.

18. *SAED* -- Selected area electron diffraction.

19. *SEM* -- Scanning electron microscope.

20. *STEM* -- Scanning transmission electron microscope.

21. *Structure* -- a microscopic bundle, cluster, fiber, or matrix which may contain asbestos.

22. *S/cm 3* -- Structures per cubic centimeter.

23. *S/mm 2* -- Structures per square millimeter.

24. *TEM* -- Transmission electron microscope. B. Sampling

1. The sampling agency must have written quality control procedures and documents which verify compliance.

2. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (References 1, 2, 3, and 5 of Unit II.J.).

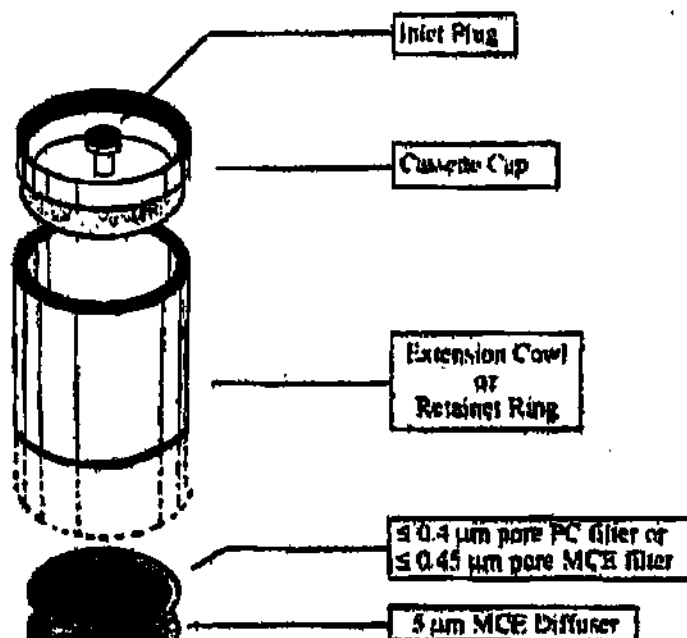
3. Sampling for airborne asbestos following an abatement action must use commercially available cassettes.

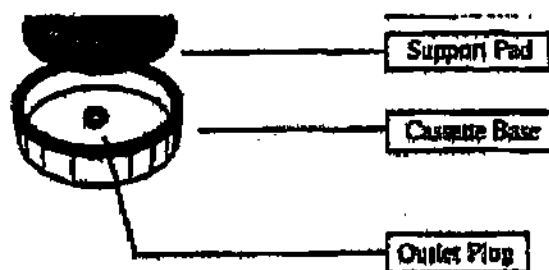
4. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm 2 in an area of 0.057 mm 2 (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm 2 for that same area is acceptable for this method.

5. Use sample collection filters which are either polycarbonate having a pore size less than or equal to 0.4  $\mu$ m or mixed cellulose ester having a pore size less than or equal to 0.45  $\mu$ m.

6. Place these filters in series with a 5.0  $\mu$ m backup filter (to serve as a diffuser) and a support pad. See the following Figure 1:

FIGURE 1--SAMPLING CASSETTE CONFIGURATION





[View or Download PDF](#)

7. Reloading of used cassettes is not permitted.
8. Orient the cassette downward at approximately 45 degrees from the horizontal.
9. Maintain a log of all pertinent sampling information.
10. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter (not the filter which will be used in sampling) before and after the sampling operation.
11. Record all calibration information.
12. Ensure that the mechanical vibrations from the pump will be minimized to prevent transferral of vibration to the cassette.
13. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by damping out any pump action fluctuations if necessary.
14. The final plastic barrier around the abatement area remains in place for the sampling period.
15. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust. (See suggested protocol in Unit III.B.7.d.)
16. Select an appropriate flow rate equal to or greater than 1 liter per minute (L/min) or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.
17. A minimum of 13 samples are to be collected for each testing site consisting of the following:
  - a. A minimum of five samples per abatement area.
  - b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.
  - c. Two field blanks are to be taken by removing the cap for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated at the following places:



- i. Near the entrance to each abatement area.
  - ii. At one of the ambient sites. (DO NOT leave the field blanks open during the sampling period.)
- d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

18. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.

19. The following Table I specifies volume ranges to be used:

TABLE I--NUMBER OF 200 MESH 25 GRID SQUARES  
(0.0025 M<sup>2</sup>) THAT NEED TO BE ANALYZED TO  
MAINTAIN SENSITIVITY OF 0.005 MICROGRAMS/CM<sup>3</sup>  
BASED ON VOLUME AND EFFECTIVE FILTER AREA

Effective Filter Area m <sup>2</sup> (sq ft)		Effective Filter Area m <sup>2</sup> (sq ft)	
Volume Range	Number of Grid Squares	Volume Range	Number of Grid Squares
100	20	1,100	10
200	10	1,200	9
300	10	1,300	9
400	7	1,400	8
500	6	1,500	8
600	6	1,600	7
700	5	1,700	7
800	5	1,800	6
900	4	1,900	6
1,000	4	2,000	5
1,100	4	2,100	5
1,200	3	2,200	4
1,300	3	2,300	4
1,400	3	2,400	4
1,500	3	2,500	3
1,600	3	2,600	3
1,700	3	2,700	3
1,800	2	2,800	3
1,900	2	2,900	2
2,000	2	3,000	2
2,100	2	3,100	2
2,200	2	3,200	2
2,300	2	3,300	2
2,400	2	3,400	2
2,500	2	3,500	2
2,600	2	3,600	2
2,700	2	3,700	2
2,800	2	3,800	2
2,900	2	3,900	2
3,000	2	4,000	2
3,100	2	4,100	2
3,200	2	4,200	2
3,300	2	4,300	2
3,400	2	4,400	2
3,500	2	4,500	2
3,600	2	4,600	2
3,700	2	4,700	2
3,800	2	4,800	2
3,900	2	4,900	2
4,000	2	5,000	2

Note: All filter volumes are based on  
a flow rate of 100 L/min  
or 2.83 m<sup>3</sup>/min

Note: All filter areas are based on a flow rate of 100 L/min  
or 2.83 m<sup>3</sup>/min

20. Ensure that the sampler is turned upright before interrupting the pump flow.
21. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.
22. Ensure that the samples are stored in a secure and representative location.
23. Do not change containers if portions of these filters are taken for other purposes.
24. A summary of Sample Data Quality Objectives is shown in the following Table II:

TABLE 31--SUMMARY OF SAMPLING ASPECTS AND QUALITY CONSIDERATIONS

This table summarizes the data quality objectives from the performance of the certified to units of precision, accuracy, repeatability, representativeness, and representability. These objectives are defined by the following units of precision and accuracy objectives listed below and described in the text of the standard.

Unit of Precision	Unit of Accuracy	Repeatability	Representativeness
Sampling results	Sample results	1 per 100 samples	95%
Sampling procedure	Field results	2 per 100 samples	95%
	Field results	Between and between field results	95%
Sample results	Representativeness (accuracy)	Field results	95% compliance
Sample shipment	Results of sampling	Field results	95% compliance

### C. Sample Shipment

Ship bulk samples to the analytical laboratory in a separate container from air samples. D. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.

2. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected. E. Sample Preparation

1. All sample preparation and analysis shall be performed by a laboratory independent of the abatement contractor.

2. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them into the clean room facility.

3. Perform sample preparation in a well-equipped clean facility.

>Note: The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA-filtered. The cumulative analytical blank concentration must average less than 18 s/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm<sup>2</sup> for that same area.

4. Preparation areas for air samples must not only be separated from preparation areas for bulk samples, but they must be prepared in separate rooms.

5. Direct preparation techniques are required. The object is to produce an intact film containing the particulates of the filter surface which is sufficiently clear for TEM analysis.

a. TEM Grid Opening Area measurement must be done as follows:

i. The filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique.

ii. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass and examining it under the PCM. Use a calibrated graticule to measure the average field

diameters. From the data, calculate the field area for an average grid opening.

iii. Measurements can also be made on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.

b. TEM specimen preparation from polycarbonate (PC) filters. Procedures as described in Unit III.G. or other equivalent methods may be used.

c. TEM specimen preparation from mixed cellulose ester (MCE) filters.

i. Filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique or the Burdette procedure (Ref. 7 of Unit II.J.)

ii. Plasma etching of the collapsed filter is required. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for the particulate asher and operating conditions will then be set such that a 1-2  $\mu\text{m}$  (10 percent) layer of collapsed surface will be removed.

iii. Procedures as described in Unit III. or other equivalent methods may be used to prepare samples.  
F. TEM Method

1. An 80-120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations is required. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely for magnification and camera constant.

2. *Determination of Camera Constant and ED Pattern Analysis.* The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulate. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter of the rings times the interplanar spacing of the ring being measured.

3. *Magnification Calibration.* The magnification calibration must be done at the fluorescent screen. The TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica (e.g., one containing 2,160

lines/mm). Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric). A logbook must be maintained, and the dates of calibration and the values obtained must be recorded. The frequency of calibration depends on the past history of the particular microscope. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate a eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.

4. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory.

5. Microscope settings: 80-120 kV, grid assessment 250-1,000X, then 15,000-20,000X screen magnification for analysis.

6. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.

7. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading must not be analyzed.

8. Reject the grid if:

- a. Less than 50 percent of the grid openings covered by the replica are intact.
- b. The replica is doubled or folded.
- c. The replica is too dark because of incomplete dissolution of the filter.

#### 9. Recording Rules.

a. Any continuous grouping of particles in which an asbestos fiber with an aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5  $\mu\text{m}$  is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. An intersection is a nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. See the following Figure 2:

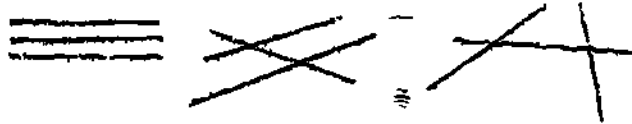
FIGURE 2.—COUNTING GUIDELINES USED IN  
DETERMINING ASBESTOS EXPOSURE

Count as 1 fiber 1 structure, no intersection.

Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or number of intersections is equal to or less than 1.



Count as 1 structure if space between fibers is greater than width of 1 fiber diameter or if the number of intersections is equal to or less than 1.

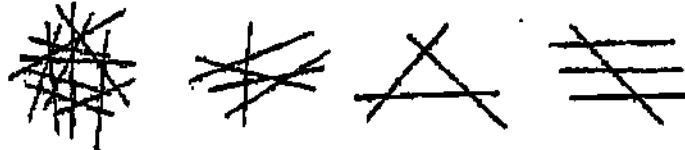


Count parallel as 1 structure; 3 or more parallel fibers less than 1 fiber diameter separation.

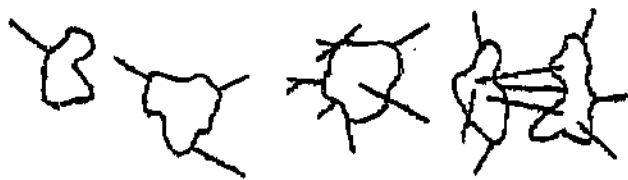


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Count clusters as 1 structure; fibers having greater than or equal to 2 intersections.



Count circles as 1 structure.



in the case of structures



Fiber processing  
 4511 Acetate Fiber

No fiber processing

Fiber processing  
 4511 Acetate Fiber

— 45.5 micrometer in length  
 — 4511 Acetate Fiber

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- i. *Fiber*. A structure having a minimum length greater than or equal to 0.5  $\mu\text{m}$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.
- ii. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- iii. *Cluster*. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.
- iv. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- b. Separate categories will be maintained for fibers less than 5  $\mu\text{m}$  and for fibers equal to or greater than 5  $\mu\text{m}$  in length.
- c. Record NSD when no structures are detected in the field.
- d. Visual identification of electron diffraction (ED) patterns is required for each asbestos structure counted which would cause the analysis to exceed the 70 s/mm<sup>2</sup> concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)
- e. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. In the event that examination of the pattern by a qualified individual indicates that the pattern has been misidentified visually, the client shall be contacted.
- f. Energy Dispersive X-ray Analysis (EDXA) is required of all amphiboles which would cause the analysis results to exceed the 70 s/mm<sup>2</sup> concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)
- g. If the number of fibers in the nonasbestos class would cause the analysis to exceed the 70 s/mm<sup>2</sup> concentration, the fact that they are not asbestos must be confirmed by EDXA or measurement of a zone axis diffraction pattern.
- h. Fibers classified as chrysotile must be identified by diffraction or X-ray analysis and recorded on a count sheet. X-ray analysis alone can be used only after 70 s/mm<sup>2</sup> have been exceeded for a particular sample.
- i. Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used only after 70 s/mm<sup>2</sup> have been exceeded for a particular sample.)
- j. If a diffraction pattern was recorded on film, record the micrograph number on the count sheet.
- k. If an electron diffraction was attempted but no pattern was observed, record N on the count sheet.

- l. If an EDXA spectrum was attempted but not observed, record N on the count sheet.
- m. If an X-ray analysis spectrum is stored, record the file and disk number on the count sheet.

#### 10. Classification Rules.

- a. *Fiber*. A structure having a minimum length greater than or equal to 0.5  $\mu\text{m}$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.
- b. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- c. *Cluster*. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.
- d. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

11. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid holder. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request. G. Sample Analytical Sequence

1. Under the present sampling requirements a minimum of 13 samples is to be collected for the clearance testing of an abatement site. These include five abatement area samples, five ambient samples, two field blanks, and one sealed blank.
2. Carry out visual inspection of work site prior to air monitoring.
3. Collect a minimum of 5 air samples inside the work site and 5 samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.

4. Remaining steps in the analytical sequence are contained in Unit IV of this Appendix. H. Reporting

1. The following information must be reported to the client for each sample analyzed:
  - a. Concentration in structures per square millimeter and structures per cubic centimeter.
  - b. Analytical sensitivity used for the analysis.
  - c. Number of asbestos structures.
  - d. Area analyzed.
  - e. Volume of air sampled (which must be initially supplied to lab by client).

- Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards are to be performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

TABLE III--SUMMARY OF LABORATORY DATA QUALITY OBJECTIVES

[illegible]

1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.
2. Check all laboratory reagents and supplies for acceptable asbestos background levels.



3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks. Testing with blanks must also be done after cleaning or servicing the room.
4. Prepare multiple grids of each sample.
5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If there are more than 53 fibers/mm<sup>2</sup> per 10 200-mesh grid openings, the system must be checked for possible sources of contamination.
6. Perform a system check on the transmission electron microscope daily.
7. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III under Unit II.I.
8. Ensure qualified operator performance by evaluation of replicate analysis and standard sample comparisons as set forth in Table III under Unit II.I.
9. Validate all data entries.
10. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III under Unit II.I.
11. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns.
12. Appropriate logs or records must be maintained by the analytical laboratory verifying that it is in compliance with the mandatory quality assurance procedures. J. References

For additional background information on this method, the following references should be consulted.

1. "Guidance for Controlling Asbestos-Containing Materials in Buildings," EPA 560/5-85-024, June 1985.
2. "Measuring Airborne Asbestos Following an Abatement Action," USEPA, Office of Pollution Prevention and Toxics, EPA 600/4-85-049, 1985.
3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.
4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjöberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.
5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4-77-027a, USEPA, Office of Research and Development, 1977.

6. Method 2A: Direct Measurement of Gas Volume through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.

7. Burdette, G.J., Health & Safety Exec. Research & Lab. Services Div., London, "Proposed Analytical Method for Determination of Asbestos in Air."

8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc., "Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters," WERL SOP 87-1, March 5, 1987.

9. NIOSH Method 7402 for Asbestos Fibers, 12-11-86 Draft.

10. Yamate, G., Agarwall, S.C., Gibbons, R.D., IIT Research Institute, "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy," Draft report, USEPA Contract 68-02-3266, July 1984.

11. "Guidance to the Preparation of Quality Assurance Project Plans," USEPA, Office of Pollution Prevention and Toxics, 1984.

### III. Nonmandatory Transmission Electron Microscopy Method

#### A. Definitions of Terms

1. *Analytical sensitivity* -- Airborne asbestos concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 s/cm<sup>3</sup>.

2. *Asbestiform* -- A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

3. *Aspect ratio* -- A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.

4. *Bundle* -- A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

5. *Clean area* -- A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200 mesh grid openings) and a maximum of 53 structures/mm<sup>2</sup> for no more than one single preparation for that same area.

6. *Cluster* -- A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

7. *ED* -- Electron diffraction.

8. *EDXA* -- Energy dispersive X-ray analysis.

9. *Fiber* -- A structure greater than or equal to 0.5  $\mu\text{m}$  in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.

10. *Grid* -- An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.

11. *Intersection* -- Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.

12. *Laboratory sample coordinator* -- That person responsible for the conduct of sample handling and the certification of the testing procedures.

13. *Filter background level* -- The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on blanks (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm<sup>2</sup>.

14. *Matrix* -- Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

15. *NSD* -- No structure detected.

16. *Operator* -- A person responsible for the TEM instrumental analysis of the sample.

17. *PCM* -- Phase contrast microscopy.

18. *SAED* -- Selected area electron diffraction.

19. *SEM* -- Scanning electron microscope.

20. *STEM* -- Scanning transmission electron microscope.

21. *Structure* -- a microscopic bundle, cluster, fiber, or matrix which may contain asbestos.

22. *S/cm<sup>3</sup>* -- Structures per cubic centimeter.

23. *S/mm<sup>2</sup>* -- Structures per square millimeter.

24. *TEM* -- Transmission electron microscope. B. Sampling

1. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (See References 1, 2, and 5 of Unit III.L.) Special precautions should be taken to avoid contamination of the sample. For example, materials that have not been prescreened for their asbestos background content should not be used; also, sample handling procedures which do not take cross contamination possibilities into account should not be used.

2. Material and supply checks for asbestos contamination should be made on all critical supplies, reagents, and procedures before their use in a monitoring study.

3. Quality control and quality assurance steps are needed to identify problem areas and isolate the cause of the contamination (see Reference 5 of Unit III.L.). Control checks shall be permanently recorded to document the quality of the information produced. The sampling firm must have written quality control procedures and documents which verify compliance. Independent audits by a qualified consultant or firm should be performed once a year. All documentation of compliance should be retained indefinitely to provide a guarantee of quality. A summary of Sample Data Quality Objectives is shown in Table II of Unit II.B.

#### 4. Sampling materials.

a. Sample for airborne asbestos following an abatement action using commercially available cassettes.

b. Use either a cowling or a filter-retaining middle piece. Conductive material may reduce the potential for particulates to adhere to the walls of the cowl.

c. Cassettes must be verified as "clean" prior to use in the field. If packaged filters are used for loading or preloaded cassettes are purchased from the manufacturer or a distributor, the manufacturer's name and lot number should be entered on all field data sheets provided to the laboratory, and are required to be listed on all reports from the laboratory.

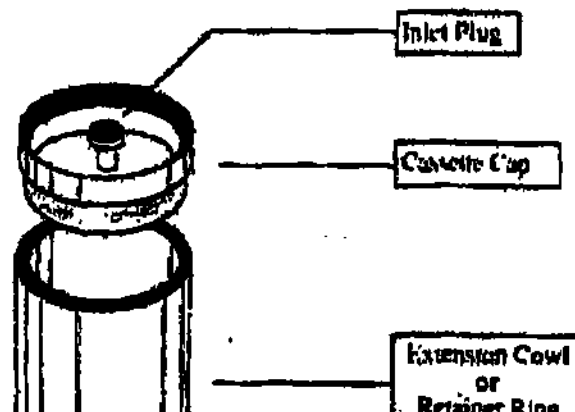
d. Assemble the cassettes in a clean facility (See definition of clean area under Unit III.A.).

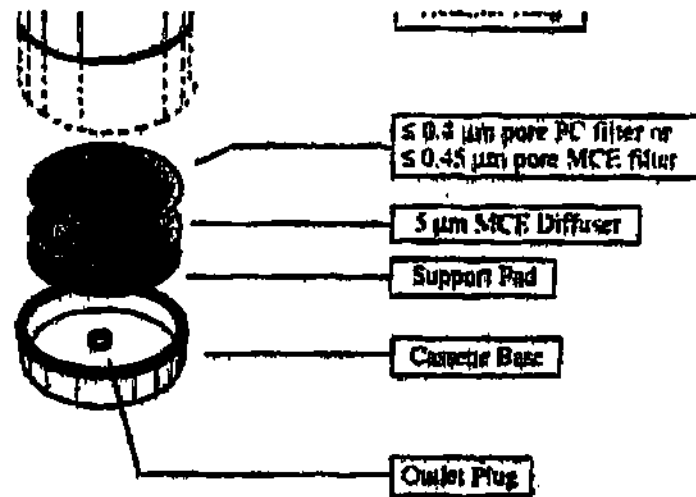
e. Reloading of used cassettes is not permitted.

f. Use sample collection filters which are either polycarbonate having a pore size of less than or equal to  $0.4\ \mu\text{m}$  or mixed cellulose ester having a pore size of less than or equal to  $0.45\ \mu\text{m}$ .

g. Place these filters in series with a backup filter with a pore size of  $5.0\ \mu\text{m}$  (to serve as a diffuser) and a support pad. See the following Figure 1:

FIGURE 1--SAMPLING CASSETTE CONFIGURATION





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h. When polycarbonate filters are used, position the highly reflective face such that the incoming particulate is received on this surface.

i. Seal the cassettes to prevent leakage around the filter edges or between cassette part joints. A mechanical press may be useful to achieve a reproducible leak-free seal. Shrink fit gel-bands may be used for this purpose and are available from filter manufacturers and their authorized distributors.

j. Use wrinkle-free loaded cassettes in the sampling operation.

#### 5. Pump setup.

a. Calibrate the sampling pump over the range of flow rates and loads anticipated for the monitoring period with this flow measuring device in series. Perform this calibration using guidance from EPA Method 2A each time the unit is sent to the field (See Reference 6 of Unit III.L.).

b. Configure the sampling system to preclude pump vibrations from being transmitted to the cassette by using a sampling stand separate from the pump station and making connections with flexible tubing.

c. Maintain continuous smooth flow conditions by damping out any pump action fluctuations if necessary.

d. Check the sampling system for leaks with the end cap still in place and the pump operating before initiating sample collection. Trace and stop the source of any flow indicated by the flowmeter under these conditions.

e. Select an appropriate flow rate equal to or greater than 1 L/min or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.

f. Orient the cassette downward at approximately 45 degrees from the horizontal.

g. Maintain a log of all pertinent sampling information, such as pump identification number, calibration data, sample location, date, sample identification number, flow rates at the beginning, middle, and end, start and stop times, and other useful information or comments. Use of a sampling log form is recommended. See the following Figure 2:

FIGURE 2--SAMPLING LOG FORM

Sample Number	Location of Sample	Pump ID	Start Time	Middle Time	End Time	Flow Rate

Inspector: \_\_\_\_\_ Date: \_\_\_\_\_

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- h. Initiate a chain of custody procedure at the start of each sampling, if this is requested by the client.
- i. Maintain a close check of all aspects of the sampling operation on a regular basis.
- j. Continue sampling until at least the minimum volume is collected, as specified in the following Table I:

TABLE 1--SUMMARY OF 200 MEAN DB EXP. DENSITIES  
(0.0057 IN<sup>3</sup>) THAT RELATE TO DB ANALYSED TO  
DETERMINE SUSCEPTIBILITY OF 0.00055 INCHES/CC  
BASED ON VOLUME AND EFFECTIVE PILEUP AREA

Relative River Area 1912-1913		Relative River Area 1913-1914	
Volume of Water CFS	Area of River Sq. Miles	Volume of Water CFS	Area of River Sq. Miles
1,000	27	1,000	27
2,000	28	1,200	28
3,000	29	1,400	29
4,000	30	1,600	30
5,000	31	1,800	31
6,000	32	2,000	32
7,000	33	2,200	33
8,000	34	2,400	34
9,000	35	2,600	35
1,000	36	2,800	36
1,100	37	3,000	37
1,200	38	3,200	38
1,300	39	3,400	39
1,400	40	3,600	40
1,500	41	3,800	41
1,600	42	4,000	42
1,700	43	4,200	43
1,800	44	4,400	44
1,900	45	4,600	45
2,000	46	4,800	46
2,100	47	5,000	47
2,200	48	5,200	48
2,300	49	5,400	49
2,400	50	5,600	50
2,500	51	5,800	51
2,600	52	6,000	52
2,700	53	6,200	53
2,800	54	6,400	54
2,900	55	6,600	55
3,000	56	6,800	56
3,100	57	7,000	57
3,200	58	7,200	58
3,300	59	7,400	59
3,400	60	7,600	60
3,500	61	7,800	61
3,600	62	8,000	62
3,700	63	8,200	63
3,800	64	8,400	64
3,900	65	8,600	65
4,000	66	8,800	66
4,100	67	9,000	67
4,200	68	9,200	68
4,300	69	9,400	69
4,400	70	9,600	70
4,500	71	9,800	71
4,600	72	10,000	72
4,700	73	10,200	73
4,800	74	10,400	74
4,900	75	10,600	75
5,000	76	10,800	76
5,100	77	11,000	77
5,200	78	11,200	78
5,300	79	11,400	79
5,400	80	11,600	80
5,500	81	11,800	81
5,600	82	12,000	82
5,700	83	12,200	83
5,800	84	12,400	84
5,900	85	12,600	85
6,000	86	12,800	86
6,100	87	13,000	87
6,200	88	13,200	88
6,300	89	13,400	89
6,400	90	13,600	90
6,500	91	13,800	91
6,600	92	14,000	92
6,700	93	14,200	93
6,800	94	14,400	94
6,900	95	14,600	95
7,000	96	14,800	96
7,100	97	15,000	97
7,200	98	15,200	98
7,300	99	15,400	99
7,400	100	15,600	100
7,500	101	15,800	101
7,600	102	16,000	102
7,700	103	16,200	103
7,800	104	16,400	104
7,900	105	16,600	105
8,000	106	16,800	106
8,100	107	17,000	107
8,200	108	17,200	108
8,300	109	17,400	109
8,400	110	17,600	110
8,500	111	17,800	111
8,600	112	18,000	112
8,700	113	18,200	113
8,800	114	18,400	114
8,900	115	18,600	115
9,000	116	18,800	116
9,100	117	19,000	117
9,200	118	19,200	118
9,300	119	19,400	119
9,400	120	19,600	120
9,500	121	19,800	121
9,600	122	20,000	122
9,700	123	20,200	123
9,800	124	20,400	124
9,900	125	20,600	125
10,000	12		

॥ श्रीगणेशाय नमः ॥  
 ॥ श्रीगणेशाय नमः ॥  
 ॥ श्रीगणेशाय नमः ॥

१. **पुस्तकालयों में २६ जून - प्रतिष्ठानों द्वारा की गई नए पुस्तकें**  
 २. **पुस्तकालयों में २७ जून - प्रतिष्ठानों द्वारा की गई नए पुस्तकें**

- k. At the conclusion of sampling, turn the cassette upward before stopping the flow to minimize possible particle loss. If the sampling is resumed, restart the flow before reorienting the cassette downward. Note the condition of the filter at the conclusion of sampling.
  - l. Double check to see that all information has been recorded on the data collection forms and that the cassette is securely closed and appropriately identified using a waterproof label. Protect cassettes in individual clean resealed polyethylene bags. Bags are to be used for storing cassette caps when they are removed for sampling purposes. Caps and plugs should only be removed or replaced using clean hands or clean disposable plastic gloves.
  - m. Do not change containers if portions of these filters are taken for other purposes.
6. Minimum sample number per site. A minimum of 13 samples are to be collected for each testing consisting of the following:
- a. A minimum of five samples per abatement area.
  - b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.
  - c. Two field blanks are to be taken by removing the cap for not more than 30 sec and replacing it at the time of sampling before sampling is initiated at the following places:

i. Near the entrance to each ambient area.

ii. At one of the ambient sites.

(Note: Do not leave the blank open during the sampling period.)

d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

#### 7. Abatement area sampling.

a. Conduct final clearance sampling only after the primary containment barriers have been removed; the abatement area has been thoroughly dried; and, it has passed visual inspection tests by qualified personnel. (See Reference 1 of Unit III.L.)

b. Containment barriers over windows, doors, and air passageways must remain in place until the TEM clearance sampling and analysis is completed and results meet clearance test criteria. The final plastic barrier remains in place for the sampling period.

c. Select sampling sites in the abatement area on a random basis to provide unbiased and representative samples.

d. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust.

i. Equipment used in aggressive sampling such as a leaf blower and/or fan should be properly cleaned and decontaminated before use.

ii. Air filtration units shall remain on during the air monitoring period.

iii. Prior to air monitoring, floors, ceiling and walls shall be swept with the exhaust of a minimum one (1) horsepower leaf blower.

iv. Stationary fans are placed in locations which will not interfere with air monitoring equipment. Fan air is directed toward the ceiling. One fan shall be used for each 10,000 ft<sup>3</sup> of worksite.

v. Monitoring of an abatement work area with high-volume pumps and the use of circulating fans will require electrical power. Electrical outlets in the abatement area may be used if available. If no such outlets are available, the equipment must be supplied with electricity by the use of extension cords and strip plug units. All electrical power supply equipment of this type must be approved Underwriter Laboratory equipment that has not been modified. All wiring must be grounded. Ground fault interrupters should be used. Extreme care must be taken to clean up any residual water and ensure that electrical equipment does not become wet while operational.

vi. Low volume pumps may be carefully wrapped in 6-mil polyethylene to insulate the pump from the air. High volume pumps cannot be sealed in this manner since the heat of the motor may melt the plastic. The pump exhausts should be kept free.



vii. If recleaning is necessary, removal of this equipment from the work area must be handled with care. It is not possible to completely decontaminate the pump motor and parts since these areas cannot be wetted. To minimize any problems in this area, all equipment such as fans and pumps should be carefully wet wiped prior to removal from the abatement area. Wrapping and sealing low volume pumps in 6-mil polyethylene will provide easier decontamination of this equipment. Use of clean water and disposable wipes should be available for this purpose.

e. Pump flow rate equal to or greater than 1 L/min or less than 10 L/min may be used for 25 mm cassettes. The larger cassette diameters may have comparably increased flow.

f. Sample a volume of air sufficient to ensure the minimum quantitation limits, (See Table I of Unit III.B.5.j.)

#### 8. Ambient sampling.

a. Position ambient samplers at locations representative of the air entering the abatement site. If makeup air entering the abatement site is drawn from another area of the building which is outside of the abatement area, place the pumps in the building, pumps should be placed out of doors located near the building and away from any obstructions that may influence wind patterns. If construction is in progress immediately outside the enclosure, it may be necessary to select another ambient site. Samples should be representative of any air entering the work site.

b. Locate the ambient samplers at least 3 ft apart and protect them from adverse weather conditions.

c. Sample same volume of air as samples taken inside the abatement site. C. Sample Shipment

1. Ship bulk samples in a separate container from air samples. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.

2. Select a rigid shipping container and pack the cassettes upright in a noncontaminating nonfibrous medium such as a bubble pack. The use of resealable polyethylene bags may help to prevent jostling of individual cassettes.

3. Avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials because of possible contamination.

4. Include a shipping bill and a detailed listing of samples shipped, their descriptions and all identifying numbers or marks, sampling data, shipper's name, and contact information. For each sample set, designate which are the ambient samples, which are the abatement area samples, which are the field blanks, and which is the sealed blank if sequential analysis is to be performed.

5. Hand-carry samples to the laboratory in an upright position if possible; otherwise choose that mode of transportation least likely to jar the samples in transit.

6. Address the package to the laboratory sample coordinator by name when known and alert him or her of the package description, shipment mode, and anticipated arrival as part of the chain of custody and sample tracking procedures. This will also help the laboratory schedule timely analysis for the samples when they are received. D. Quality Control/Quality Assurance Procedures (Data Quality

**Indicators)**

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined, and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the text below.

1. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200-mesh grid openings) and a maximum of 53 s/mm<sup>2</sup> for that same area for any single preparation is acceptable for this method.
2. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter -- not the filter which will be used in sampling -- before and after the sampling operation.
3. Record all calibration information with the data to be used on a standard sampling form.
4. Ensure that the samples are stored in a secure and representative location.
5. Ensure that mechanical calibrations from the pump will be minimized to prevent transferral of vibration to the cassette.
6. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by installing a damping chamber if necessary.
7. Open a loaded cassette momentarily at one of the indoor sampling sites when sampling is initiated. This sample will serve as an indoor field blank.
8. Open a loaded cassette momentarily at one of the outdoor sampling sites when sampling is initiated. This sample will serve as an outdoor field blank.
9. Carry a sealed blank into the field with each sample series. Do not open this cassette in the field.
10. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.
11. Ensure that the sampler is turned upright before interrupting the pump flow.
12. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory. E. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.
2. Adhere to the following procedures to ensure both the continued chain-of-custody and the accountability of all samples passing through the laboratory:
  - a. Note the condition of the shipping package and data written on it upon receipt.
  - b. Retain all bills of lading or shipping slips to document the shipper and delivery time.
  - c. Examine the chain-of-custody seal, if any, and the package for its integrity.
  - d. If there has been a break in the seal or substantive damage to the package, the sample coordinator shall immediately notify the shipper and a responsible laboratory manager before any action is taken to unpack the shipment.
  - e. Packages with significant damage shall be accepted only by the responsible laboratory manager after discussions with the client.
3. Unwrap the shipment in a clean, uncluttered facility. The sample coordinator or his or her designee will record the contents, including a description of each item and all identifying numbers or marks. A Sample Receiving Form to document this information is attached for use when necessary. (See the following Figure 3.)

FIGURE 3--SAMPLE RECEIVING FORM

Date of package delivery _____	Package shipped from _____
Carrier _____	Shipping bill number _____
*Condition of package on receipt _____	
*Condition of custody seal _____	
Number of samples received _____	Shipping manifest attached _____
Package Order No. _____	Package ID# _____
Comments: _____	

No.	Description	Sampling Location	Sampling Date	Sampling Time	Remarks
1	_____	_____	_____	_____	_____
2	_____	_____	_____	_____	_____
3	_____	_____	_____	_____	_____
4	_____	_____	_____	_____	_____
5	_____	_____	_____	_____	_____
6	_____	_____	_____	_____	_____
7	_____	_____	_____	_____	_____
8	_____	_____	_____	_____	_____
9	_____	_____	_____	_____	_____
10	_____	_____	_____	_____	_____
11	_____	_____	_____	_____	_____
12	_____	_____	_____	_____	_____
13	_____	_____	_____	_____	_____
14	_____	_____	_____	_____	_____

(Use as many additional sheets as needed.)

Comments: \_\_\_\_\_

Date of sample receipt \_\_\_\_\_

Signature of chain-of-custody recipient \_\_\_\_\_

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**Note:** The person breaking the chain-of-custody seal and itemizing the contents assumes responsibility for the shipment and signs documents accordingly.

4. Assign a laboratory number and schedule an analysis sequence.

5. Manage all chain-of-custody samples within the laboratory such that their integrity can be ensured and documented. F. Sample Preparation

1. Personnel not affiliated with the Abatement Contractor shall be used to prepare samples and conduct TEM analysis. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them to the clean sample preparation facility.

2. Perform sample preparation in a well-equipped clean facility.

**Note:** The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA filtered. The cumulative analytical blank concentration must average less than 18 s/mm<sup>2</sup> in an area of 0.057 s/mm<sup>2</sup> (nominally 10 200-mesh grid openings) with no more than one single preparation to exceed 53 s/mm<sup>2</sup> for that same area.

3. Preparation areas for air samples must be separated from preparation areas for bulk samples. Personnel must not prepare air samples if they have previously been preparing bulk samples without performing appropriate personal hygiene procedures, i.e., clothing change, showering, etc.

4. *Preparation.* Direct preparation techniques are required. The objective is to produce an intact carbon film containing the particulates from the filter surface which is sufficiently clear for TEM analysis. Currently recommended direct preparation procedures for polycarbonate (PC) and mixed cellulose ester (MCE) filters are described in Unit III.F.7. and 8. Sample preparation is a subject requiring additional research. Variation on those steps which do not substantively change the procedure, which improve filter clearing or which reduce contamination problems in a laboratory are permitted.

a. Use only TEM grids that have had grid opening areas measured according to directions in Unit III.J.

b. Remove the inlet and outlet plugs prior to opening the cassette to minimize any pressure differential that may be present.

c. Examples of techniques used to prepare polycarbonate filters are described in Unit III.F.7.

d. Examples of techniques used to prepare mixed cellulose ester filters are described in Unit III.F.8.

e. Prepare multiple grids for each sample.

f. Store the three grids to be measured in appropriately labeled grid holders or polyethylene capsules.

## 5. Equipment.

a. Clean area.

b. Tweezers. Fine-point tweezers for handling of filters and TEM grids.

c. Scalpel Holder and Curved No. 10 Surgical Blades.

d. Microscope slides.

e. Double-coated adhesive tape.

f. Gummed page reinforcements.

g. Micro-pipet with disposal tips 10 to 100  $\mu$ L variable volume.

h. Vacuum coating unit with facilities for evaporation of carbon. Use of a liquid nitrogen cold trap above the diffusion pump will minimize the possibility of contamination of the filter surface by oil from the pumping system. The vacuum-coating unit can also be used for deposition of a thin film of gold.

i. *Carbon rod electrodes*. Spectrochemically pure carbon rods are required for use in the vacuum evaporator for carbon coating of filters.

j. *Carbon rod sharpener*. This is used to sharpen carbon rods to a neck. The use of necked carbon rods (or equivalent) allows the carbon to be applied to the filters with a minimum of heating.

k. *Low-temperature plasma asher*. This is used to etch the surface of collapsed mixed cellulose ester (MCE) filters. The asher should be supplied with oxygen, and should be modified as necessary to provide a throttle or bleed valve to control the speed of the vacuum to minimize disturbance of the filter. Some early models of ashers admit air too rapidly, which may disturb particulates on the surface of the filter during the etching step.

l. *Glass petri dishes, 10 cm in diameter, 1 cm high*. For prevention of excessive evaporation of solvent when these are in use, a good seal must be provided between the base and the lid. The seal can be improved by grinding the base and lid together with an abrasive grinding material.

m. Stainless steel mesh.

n. Lens tissue.

o. Copper 200-mesh TEM grids, 3 mm in diameter, or equivalent.

p. Gold 200-mesh TEM grids, 3 mm in diameter, or equivalent.

- q. Condensation washer.
- r. Carbon-coated, 200-mesh TEM grids, or equivalent.
- s. Analytical balance, 0.1 mg sensitivity.
- t. Filter paper, 9 cm in diameter.
- u. Oven or slide warmer. Must be capable of maintaining a temperature of 65-70 °C.
- v. Polyurethane foam, 6 mm thickness.
- w. Gold wire for evaporation.

#### 6. Reagents.

a. *General.* A supply of ultra-clean, fiber-free water must be available for washing of all components used in the analysis. Water that has been distilled in glass or filtered or deionized water is satisfactory for this purpose. Reagents must be fiber-free.

b. Polycarbonate preparation method -- chloroform.

c. Mixed Cellulose Ester (MCE) preparation method -- acetone or the Burdette procedure (Ref. 7 of Unit III.L.).

#### 7. TEM specimen preparation from polycarbonate filters.

a. *Specimen preparation laboratory.* It is most important to ensure that contamination of TEM specimens by extraneous asbestos fibers is minimized during preparation.

b. *Cleaning of sample cassettes.* Upon receipt at the analytical laboratory and before they are taken into the clean facility or laminar flow hood, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces.

c. *Preparation of the carbon evaporator.* If the polycarbonate filter has already been carbon-coated prior to receipt, the carbon coating step will be omitted, unless the analyst believes the carbon film is too thin. If there is a need to apply more carbon, the filter will be treated in the same way as an uncoated filter. Carbon coating must be performed with a high-vacuum coating unit. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application, and must not be used. The carbon rods should be sharpened by a carbon rod sharpener to necks of about 4 mm long and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 10 to 12 cm from the surface of a microscope slide held in the rotating and tilting device.

d. *Selection of filter area for carbon coating.* Before preparation of the filters, a 75 mm x 50 mm microscope slide is washed and dried. This slide is used to support strips of filter during the carbon evaporation. Two parallel strips of double-sided adhesive tape are applied along the length of the slide. Polycarbonate filters are easily stretched during handling, and cutting of areas for further

preparation must be performed with great care. The filter and the MCE backing filter are removed together from the cassette and placed on a cleaned glass microscope slide. The filter can be cut with a curved scalpel blade by rocking the blade from the point placed in contact with the filter. The process can be repeated to cut a strip approximately 3 mm wide across the diameter of the filter. The strip of polycarbonate filter is separated from the corresponding strip of backing filter and carefully placed so that it bridges the gap between the adhesive tape strips on the microscope slide. The filter strip can be held with fine-point tweezers and supported underneath by the scalpel blade during placement on the microscope slide. The analyst can place several such strips on the same microscope slide, taking care to rinse and wet-wipe the scalpel blade and tweezers before handling a new sample. The filter strips should be identified by etching the glass slide or marking the slide using a marker insoluble in water and solvents. After the filter strip has been cut from each filter, the residual parts of the filter must be returned to the cassette and held in position by reassembly of the cassette. The cassette will then be archived for a period of 30 days or returned to the client upon request.

e. **Carbon coating of filter strips.** The glass slide holding the filter strips is placed on the rotation-tilting device, and the evaporator chamber is evacuated. The evaporation must be performed in very short bursts, separated by some seconds to allow the electrodes to cool. If evaporation is too rapid, the strips of polycarbonate filter will begin to curl, which will lead to cross-linking of the surface material and make it relatively insoluble in chloroform. An experienced analyst can judge the thickness of carbon film to be applied, and some test should be made first on unused filters. If the film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen. If the coating is too thick, the filter will tend to curl when exposed to chloroform vapor and the carbon film may not adhere to the support mesh. Too thick a carbon film will also lead to a TEM image that is lacking in contrast, and the ability to obtain ED patterns will be compromised. The carbon film should be as thin as possible and remain intact on most of the grid openings of the TEM specimen intact.

f. **Preparation of the Jaffe washer.** The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used. A washer consisting of a simple stainless steel bridge is recommended. Several pieces of lens tissue approximately 1.0 cm x 0.5 cm are placed on the stainless steel bridge, and the washer is filled with chloroform to a level where the meniscus contacts the underside of the mesh, which results in saturation of the lens tissue. See References 8 and 10 of Unit III.L.

g. **Placing of specimens into the Jaffe washer.** The TEM grids are first placed on a piece of lens tissue so that individual grids can be picked up with tweezers. Using a curved scalpel blade, the analyst excises three 3 mm square pieces of the carbon-coated polycarbonate filter from the filter strip. The three squares are selected from the center of the strip and from two points between the outer periphery of the active surface and the center. The piece of filter is placed on a TEM specimen grid with the shiny side of the TEM grid facing upwards, and the whole assembly is placed boldly onto the saturated lens tissue in the Jaffe washer. If carbon-coated grids are used, the filter should be placed carbon-coated side down. The three excised squares of filters are placed on the same piece of lens tissue. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. The lid is then placed on the Jaffe washer, and the system is allowed to stand for several hours, preferably overnight.

h. **Condensation washing.** It has been found that many polycarbonate filters will not dissolve completely in the Jaffe washer, even after being exposed to chloroform for as long as 3 days. This problem becomes more serious if the surface of the filter was overheated during the carbon

evaporation. The presence of undissolved filter medium on the TEM preparation leads to partial or complete obscuration of areas of the sample, and fibers that may be present in these areas of the specimen will be overlooked; this will lead to a low result. Undissolved filter medium also compromises the ability to obtain ED patterns. Before they are counted, TEM grids must be examined critically to determine whether they are adequately cleared of residual filter medium. It has been found that condensation washing of the grids after the initial Jaffe washer treatment, with chloroform as the solvent, clears all residual filter medium in a period of approximately 1 hour. In practice, the piece of lens tissue supporting the specimen grids is transferred to the cold finger of the condensation washer, and the washer is operated for about 1 hour. If the specimens are cleared satisfactorily by the Jaffe washer alone, the condensation washer step may be unnecessary.

#### 8. TEM specimen preparation from MCE filters.

a. This method of preparing TEM specimens from MCE filters is similar to that specified in NIOSH Method 7402. See References 7, 8, and 9 of Unit III.L.

b. Upon receipt at the analytical laboratory, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces before entering the clean sample preparation area.

c. Remove a section from any quadrant of the sample and blank filters.

d. Place the section on a clean microscope slide. Affix the filter section to the slide with a gummed paged reinforcement or other suitable means. Label the slide with a water and solvent-proof marking pen.

e. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 minutes for the sample filter to fuse and clear.

f. Plasma etching of the collapsed filter is required.

i. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. This is one area of the method that requires further evaluation. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for a particular asher and operating conditions will then be set such that a 1-2  $\mu\text{m}$  (10 percent) layer of collapsed surface will be removed.

ii. Place the slide containing the collapsed filters into a low-temperature plasma asher, and etch the filter.

g. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1 mm x 5 mm section of graphite rod onto the cleared filter. Remove the slide to a clean, dry, covered petri dish.

h. Prepare a second petri dish as a Jaffe washer with the wicking substrate prepared from filter or lens paper placed on top of a 6 mm thick disk of clean spongy polyurethane foam. Cut a V-notch on the



edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent. The wicking substrate should be thin enough to fit into the petri dish without touching the lid.

i. Place carbon-coated TEM grids face up on the filter or lens paper. Label the grids by marking with a pencil on the filter paper or by putting registration marks on the petri dish lid and marking with a waterproof marker on the dish lid. In a fume hood, fill the dish with acetone until the wicking substrate is saturated. The level of acetone should be just high enough to saturate the filter paper without creating puddles.

j. Remove about a quarter section of the carbon-coated filter samples from the glass slides using a surgical knife and tweezers. Carefully place the section of the filter, carbon side down, on the appropriately labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to bring the acetone level up to the highest possible level without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it. This allows drops of condensed solvent vapors to form near the edge rather than in the center where they would drip onto the grid preparation. G. TEM Method

#### 1. Instrumentation.

a. Use an 80-120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely (see Unit III.J.) for magnification and camera constant.

b. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory. This must be an Energy Dispersive X-ray Detector mounted on TEM column and associated hardware/software to collect, save, and read out spectral information. Calibration of Multi-Channel Analyzer shall be checked regularly for Al at 1.48 KeV and Cu at 8.04 KeV, as well as the manufacturer's procedures.

i. Standard replica grating may be used to determine magnification (e.g., 2160 lines/mm).

ii. Gold standard may be used to determine camera constant.

c. Use a specimen holder with single tilt and/or double tilt capabilities.

#### 2. Procedure.

a. Start a new Count Sheet for each sample to be analyzed. Record on count sheet; analyst's initials and date; lab sample number; client sample number microscope identification; magnification for analysis; number of predetermined grid openings to be analyzed; and grid identification. See the following Figure 4:

[illegible]

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- b. Check that the microscope is properly aligned and calibrated according to the manufacturer's specifications and instructions.
- c. Microscope settings: 80-120 kV, grid assessment 250-1000X, then 15,000-20,000X screen magnification for analysis.
- d. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.
- e. Determine the suitability of the grid.
  - i. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading shall not be analyzed.
  - ii. Examine the grid at low magnification (<1000X) to determine its suitability for detailed study at higher magnifications.
  - iii. Reject the grid if:

(1) Less than 50 percent of the grid openings covered by the replica are intact.

(2) It is doubled or folded.

(3) It is too dark because of incomplete dissolution of the filter.

iv. If the grid is rejected, load the next sample grid.

v. If the grid is acceptable, continue on to Step 6 if mapping is to be used; otherwise proceed to Step 7.

f. Grid Map (Optional).

i. Set the TEM to the low magnification mode.

ii. Use flat edge or finder grids for mapping.

iii. Index the grid openings (fields) to be counted by marking the acceptable fields for one-half (0.5) of the area needed for analysis on each of the two grids to be analyzed. These may be marked just before examining each grid opening (field), if desired.

iv. Draw in any details which will allow the grid to be properly oriented if it is reloaded into the microscope and a particular field is to be reliably identified.

g. Scan the grid.

i. Select a field to start the examination.

ii. Choose the appropriate magnification (15,000 to 20,000X screen magnification).

iii. Scan the grid as follows.

(1) At the selected magnification, make a series of parallel traverses across the field. On reaching the end of one traverse, move the image one window and reverse the traverse.

**Note:** A slight overlap should be used so as not to miss any part of the grid opening (field).

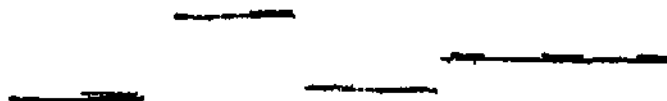
(2) Make parallel traverses until the entire grid opening (field) has been scanned.

h. Identify each structure for appearance and size.

i. Appearance and size: Any continuous grouping of particles in which an asbestos fiber within aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5  $\mu\text{m}$  is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. See the following Figure 5:

FIGURE 1. CLASSIFICATION OF FIBER STRUCTURES  
 DEFINITION: ASSEMBLED STRUCTURES

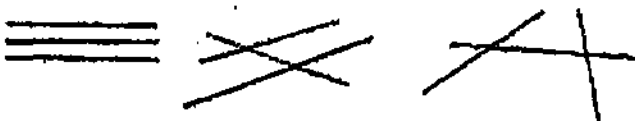
Count as 1 cluster, 1 structure; no intersections.



Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or radius of intersection is equal to or less than 1.



Count as 3 structures if space between fibers is greater than width of 1 fiber diameter or if the radius of intersection is equal to or less than 2.

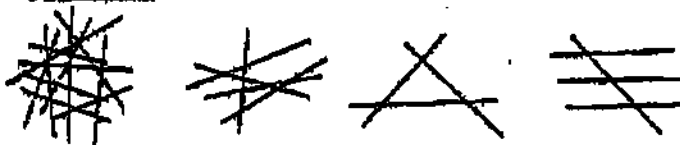


Count bundles as 1 structure; 3 or more parallel fibers less than 1 fiber diameter apart are.

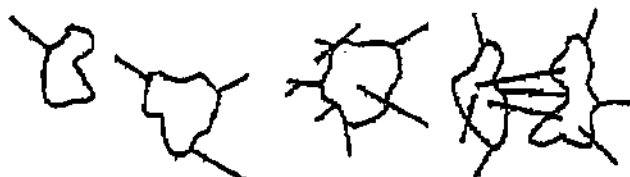


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Count clusters as 1 structure; fibers having greater than or equal to 3 intersections.



Count groups as 1 structure.



Do not count as structures:



Fiber protrusion  
 does not count

No fiber protrusion

Fiber Properties  
 <0.5 micrometers

<0.5 micrometers in length  
 5:1 Aspect Ratio

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An intersection is a non-parallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. Combinations such as a matrix and cluster, matrix and bundle, or bundle and cluster are categorized by the dominant fiber quality — cluster, bundle, and matrix, respectively. Separate categories will be maintained for fibers less than 5  $\mu\text{m}$  and for fibers greater than or equal to 5  $\mu\text{m}$  in length. Not required, but useful, may be to record the fiber length in 1  $\mu\text{m}$  intervals. (Identify each structure morphologically and analyze it as it enters the "window".)

- (1) *Fiber*. A structure having a minimum length greater than 0.5  $\mu\text{m}$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed, no intersections.
- (2) *Bundle*. A structure composed of 3 or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- (3) *Cluster*. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings must have more than 2 intersections.
- (4) *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- (5) *NSD*. Record NSD when no structures are detected in the field.
- (6) *Intersection*. Non-parallel touching or crossing of fibers, with the projection having an aspect ratio 5:1 or greater.

## ii. Structure Measurement.

- (1) Recognize the structure that is to be sized.
- (2) Memorize its location in the "window" relative to the sides, inscribed square and to other particulates in the field so this exact location can be found again when scanning is resumed.
- (3) Measure the structure using the scale on the screen.
- (4) Record the length category and structure type classification on the count sheet after the field number and fiber number.
- (5) Return the fiber to its original location in the window and scan the rest of the field for other fibers; if the direction of travel is not remembered, return to the right side of the field and begin the traverse again.

i. Visual identification of Electron Diffraction (ED) patterns is required for each asbestos structure counted which would cause the analysis to exceed the 70 s/mm<sup>2</sup> concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)

i. Center the structure, focus, and obtain an ED pattern. (See Microscope Instruction Manual for more detailed instructions.)

ii. From a visual examination of the ED pattern, obtained with a short camera length, classify the observed structure as belonging to one of the following classifications: chrysotile, amphibole, or nonasbestos.

(1) Chrysotile: The chrysotile asbestos pattern has characteristic streaks on the layer lines other than the central line and some streaking also on the central line. There will be spots of normal sharpness on the central layer line and on alternate lines (2nd, 4th, etc.). The repeat distance between layer lines is 0.53 nm and the center doublet is at 0.73 nm. The pattern should display (002), (110), (130) diffraction maxima; distances and geometry should match a chrysotile pattern and be measured semiquantitatively.

(2) Amphibole Group [includes grunerite (amosite), crocidolite, anthophyllite, tremolite, and actinolite]: Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and the repeat distance between layer lines is also about 0.53 nm. Streaking in layer lines is occasionally present due to crystal structure defects.

(3) Nonasbestos: Incomplete or unobtainable ED patterns, a nonasbestos EDXA, or a nonasbestos morphology.

iii. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns. In the event that examination of the pattern by the qualified individual indicates that the pattern had been misidentified visually, the client shall be contacted. If the pattern is a suspected chrysotile, take a photograph of the diffraction pattern at 0 degrees tilt. If the structure is suspected to be amphibole, the sample may have to be tilted to obtain a simple geometric array of spots.

#### j. Energy Dispersive X-Ray Analysis (EDXA).

i. Required of all amphiboles which would cause the analysis results to exceed the 70 s/mm<sup>2</sup> concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)

ii. Can be used alone to confirm chrysotile after the 70 s/mm<sup>2</sup> concentration has been exceeded.

iii. Can be used alone to confirm all nonasbestos.

iv. Compare spectrum profiles with profiles obtained from asbestos standards. The closest match identifies and categorizes the structure.

v. If the EDXA is used for confirmation, record the properly labeled spectrum on a computer disk, or if a hard copy, file with analysis data.

vi. If the number of fibers in the nonasbestos class would cause the analysis to exceed the 70 s/mm<sup>2</sup> concentration, their identities must be confirmed by EDXA or measurement of a zone axis diffraction pattern to establish that the particles are nonasbestos.

**k. Stopping Rules.**

i. If more than 50 asbestiform structures are counted in a particular grid opening, the analysis may be terminated.

ii. After having counted 50 asbestiform structures in a minimum of 4 grid openings, the analysis may be terminated. The grid opening in which the 50th fiber was counted must be completed.

iii. For blank samples, the analysis is always continued until 10 grid openings have been analyzed.

iv. In all other samples the analysis shall be continued until an analytical sensitivity of 0.005 s/cm<sup>3</sup> is reached.

**l. Recording Rules.** The count sheet should contain the following information:

i. Field (grid opening): List field number.

ii. Record "NSD" if no structures are detected.

iii. Structure information.

(1) If fibers, bundles, clusters, and/or matrices are found, list them in consecutive numerical order, starting over with each field.

(2) Length. Record length category of asbestos fibers examined. Indicate if less than 5  $\mu$ m or greater than or equal to 5  $\mu$ m.

(3) Structure Type. Positive identification of asbestos fibers is required by the method. At least one diffraction pattern of each fiber type from every five samples must be recorded and compared with a standard diffraction pattern. For each asbestos fiber reported, both a morphological descriptor and an identification descriptor shall be specified on the count sheet.

(4) Fibers classified as chrysotile must be identified by diffraction and/or X-ray analysis and recorded on the count sheet. X-ray analysis alone can be used as sole identification only after 70s/mm<sup>2</sup> have been exceeded for a particular sample.

(5) Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used as sole identification only after 70s/mm<sup>2</sup> have been exceeded for a particular sample.)

(6) If a diffraction pattern was recorded on film, the micrograph number must be indicated on the

count sheet.

(7) If an electron diffraction was attempted and an appropriate spectra is not observed, N should be recorded on the count sheet.

(8) If an X-ray analysis is attempted but not observed, N should be recorded on the count sheet.

(9) If an X-ray analysis spectrum is stored, the file and disk number must be recorded on the count sheet.

#### m. Classification Rules.

i. *Fiber*. A structure having a minimum length greater than or equal to 0.5  $\mu\text{m}$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

ii. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

iii. *Cluster*. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

iv. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

v. *NSD*. Record NSD when no structures are detected in the field.

n. After all necessary analyses of a particle structure have been completed, return the goniometer stage to 0 degrees, and return the structure to its original location by recall of the original location.

o. Continue scanning until all the structures are identified, classified and sized in the field.

p. Select additional fields (grid openings) at low magnification; scan at a chosen magnification (15,000 to 20,000X screen magnification); and analyze until the stopping rule becomes applicable.

q. Carefully record all data as they are being collected, and check for accuracy.

r. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid hold. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request. H. Sample Analytical Sequence

1. Carry out visual inspection of work site prior to air monitoring.

2. Collect a minimum of five air samples inside the work site and five samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.

3. Analyze the abatement area samples according to this protocol. The analysis must meet the 0.005 s/cm 3 analytical sensitivity.



**4. Remaining steps in the analytical sequence are contained in Unit IV. of this Appendix. I. Reporting**

**The following information must be reported to the client. See the following Table II:**

**EXHIBIT 12--EXAMPLE LABORATORY LETTERHEAD**

[illegible]

### INDIVIDUAL ANALYTICAL RESULTS

[illegible]

The analysis was carried out in the approved TEM method. This laboratory is in compliance with the guidelines specified by the method.

## **Abstract**

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1. Concentration in structures per square millimeter and structures per cubic centimeter.
2. Analytical sensitivity used for the analysis.
3. Number of asbestos structures.
4. Area analyzed.
5. Volume of air samples (which was initially provided by client).
6. Average grid size opening.
7. Number of grids analyzed.
8. Copy of the count sheet must be included with the report.

9. Signature of laboratory official to indicate that the laboratory met specifications of the AHERA method.

10. Report form must contain official laboratory identification (e.g., letterhead).

#### 11. Type of asbestos. J. Calibration Methodology

**Note:** Appropriate implementation of the method requires a person knowledgeable in electron diffraction and mineral identification by ED and EDXA. Those inexperienced laboratories wishing to develop capabilities may acquire necessary knowledge through analysis of appropriate standards and by following detailed methods as described in References 8 and 10 of Unit III.L.

1. *Equipment Calibration.* In this method, calibration is required for the air-sampling equipment and the transmission electron microscope (TEM).

a. *TEM Magnification.* The magnification at the fluorescent screen of the TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica. A logbook must be maintained, and the dates of calibration depend on the past history of the particular microscope; no frequency is specified. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate an eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.

b. Determination of the TEM magnification on the fluorescent screen.

i. Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

ii. Insert a diffraction grating replica (for example a grating containing 2,160 lines/mm) into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that the goniometer stage tilt is 0 degrees.

iii. Adjust microscope magnification to 10,000X or 20,000X. Measure the distance (mm) between two widely separated lines on the grating replica. Note the number of spaces between the lines. Take care to measure between the same relative positions on the lines (e.g., between left edges of lines).

**Note:** The more spaces included in the measurement, the more accurate the final calculation. On most microscopes, however, the magnification is substantially constant only within the central 8-10 cm diameter region of the fluorescent screen.

iv. Calculate the true magnification (M) on the fluorescent screen:

11-XXXX

VI-AU/ I

where:

$X$ =total distance (mm) between the designated grating lines;

$G$ =calibration constant of the grating replica (lines/mm);

$Y$ =number of grating replica spaces counted along  $X$ .

c. Calibration of the EDXA System. Initially, the EDXA system must be calibrated by using two reference elements to calibrate the energy scale of the instrument. When this has been completed in accordance with the manufacturer's instructions, calibration in terms of the different types of asbestos can proceed. The EDXA detectors vary in both solid angle of detection and in window thickness. Therefore, at a particular accelerating voltage in use on the TEM, the count rate obtained from specific dimensions of fiber will vary both in absolute X-ray count rate and in the relative X-ray peak heights for different elements. Only a few minerals are relevant for asbestos abatement work, and in this procedure the calibration is specified in terms of a "fingerprint" technique. The EDXA spectra must be recorded from individual fibers of the relevant minerals, and identifications are made on the basis of semiquantitative comparisons with these reference spectra.

#### d. Calibration of Grid Openings.

i. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass slide and examining it under the PCM. Use a calibrated graticule to measure the average field diameter and use this number to calculate the field area for an average grid opening. Grids are to be randomly selected from batches up to 1,000.

Note: A grid opening is considered as one field.

ii. The mean grid opening area must be measured for the type of specimen grids in use. This can be accomplished on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.

#### e. Determination of Camera Constant and ED Pattern Analysis.

i. The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film.

ii. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulates. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one half the diameter

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

[illegible]

- .../otfilter.cgi?DB=1&ACTION=View&OUERY=40&RGN=BTI&OP=and&OUERY=763&R7/16/2000

If this average is greater than 0.1 mm / 2 per 10 200-mesh grid openings, check the system for possible sources of contamination.

6. Check for recovery of asbestos from cellulose ester filters submitted to plasma asher.
7. Check for asbestos carryover in the plasma asher by including a blank alongside the positive control sample.
8. Perform a systems check on the transmission electron microscope daily.
9. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III of Unit III.K.
10. Ensure qualified operator performance by evaluation of replicate counting, duplicate analysis, and standard sample comparisons as set forth in Table III of Unit III.K.
11. Validate all data entries.
12. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III.
13. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions. The outline of quality control procedures presented above is viewed as the minimum required to assure that quality data is produced for clearance testing of an asbestos abated area. Additional information may be gained by other control tests. Specifics on those control procedures and options available for environmental testing can be obtained by consulting References 6, 7, and 11 of Unit III.L. L. References

For additional background information on this method the following references should be consulted.

1. "Guidelines for Controlling Asbestos-Containing Materials in Buildings," EPA 560/5-85-024, June 1985.
2. "Measuring Airborne Asbestos Following an Abatement Action," USEP/Office of Pollution Prevention and Toxics, EPA 600/4-85-049, 1985.
3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.
4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjöberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.
5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4-77-027a, USEPA, Office of Research and Development, 1977.
6. Method 2A: Direct Measurement of Gas Volume Through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.

7. Handbook of Asbestos and Asbestos-Containing Materials. Environmental Protection Agency, Washington, D.C., 1977.

7. Burdett, G.J. Health & Safety Exec., Research & Lab. Services Div., London, "Proposed Analytical Method for Determination of Asbestos in Air."

8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc. "Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters." WERL SOP 87-1, March 5, 1987.

9. NIOSH. Method 7402 for Asbestos Fibers, December 11, 1986 Draft.

10. Yamate, G., S.C. Agarwall, R.D. Gibbons, IIT Research Institute, "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy." Draft report, USEPA Contract 68-02-3266, July 1984.

11. Guidance to the Preparation of Quality Assurance Project Plans. USEPA, Office of Pollution Prevention and Toxics, 1984.

#### IV. Mandatory Interpretation of Transmission Electron Microscopy Results to Determine Completion of Response Actions

##### A. Introduction

A response action is determined to be completed by TEM when the abatement area has been cleaned and the airborne asbestos concentration inside the abatement area is no higher than concentrations at locations outside the abatement area. "Outside" means outside the abatement area, but not necessarily outside the building. EPA reasons that an asbestos removal contractor cannot be expected to clean an abatement area to an airborne asbestos concentration that is lower than the concentration of air entering the abatement area from outdoors or from other parts of the building. After the abatement area has passed a thorough visual inspection, and before the outer containment barrier is removed, a minimum of five air samples inside the abatement area and a minimum of five air samples outside the abatement area must be collected. Hence, the response action is determined to be completed when the average airborne asbestos concentration measured inside the abatement area is not statistically different from the average airborne asbestos concentration measured outside the abatement area.

The inside and outside concentrations are compared by the Z-test, a statistical test that takes into account the variability in the measurement process. A minimum of five samples inside the abatement area and five samples outside the abatement area are required to control the false negative error rate, i.e., the probability of declaring the removal complete when, in fact, the air concentration inside the abatement area is significantly higher than outside the abatement area. Additional quality control is provided by requiring three blanks (filters through which no air has been drawn) to be analyzed to check for unusually high filter contamination that would distort the test results.

When volumes greater than or equal to 1,199 L for a 25 mm filter and 2,799 L for a 37 mm filter have been collected and the average number of asbestos structures on samples inside the abatement area is no greater than 70 s/mm<sup>2</sup> of filter, the response action may be considered complete without comparing the inside samples to the outside samples. EPA is permitting this initial screening test to save analysis costs in situations where the airborne asbestos concentration is sufficiently low so that it cannot be distinguished from the filter contamination/background level (fibers deposited on the filter that are unrelated to the air being sampled). The screening test cannot be used when volumes of less than 1,199 L for 25 mm filter or 2,799 L for a 37 mm filter are collected because the ability to distinguish levels significantly different from filter background is

reduced at low volumes.

The initial screening test is expressed in structures per square millimeter of filter because filter background levels come from sources other than the air being sampled and cannot be meaningfully expressed as a concentration per cubic centimeter of air. The value of 70 s/mm<sup>2</sup> is based on the experience of the panel of microscopists who consider one structure in 10 grid openings (each grid opening with an area of 0.0057 mm<sup>2</sup>) to be comparable with contamination/background levels of blank filters. The decision is based, in part, on Poisson statistics which indicate that four structures must be counted on a filter before the fiber count is statistically distinguishable from the count for one structure. As more information on the performance of the method is collected, this criterion may be modified. Since different combinations of the number and size of grid openings are permitted under the TEM protocol, the criterion is expressed in structures per square millimeter of filter to be consistent across all combinations. Four structures per 10 grid openings corresponds to approximately 70 s/mm<sup>2</sup>. B. Sample Collection and Analysis

1. A minimum of 13 samples is required: five samples collected inside the abatement area, five samples collected outside the abatement area, two field blanks, and one sealed blank.
2. Sampling and TEM analysis must be done according to either the mandatory or nonmandatory protocols in Appendix A. At least 0.057 mm<sup>2</sup> of filter must be examined on blank filters. C. Interpretation of Results

1. The response action shall be considered complete if either:

- a. Each sample collected inside the abatement area consists of at least 1,199 L of air for a 25 mm filter, or 2,799 L of air for a 37 mm filter, and the arithmetic mean of their asbestos structure concentrations per square millimeter of filter is less than or equal to 70 s/mm<sup>2</sup>; or
- b. The three blank samples have an arithmetic mean of the asbestos structure concentration on the blank filters that is less than or equal to 70 s/mm<sup>2</sup> and the average airborne asbestos concentration measured inside the abatement area is not statistically higher than the average airborne asbestos concentration measured outside the abatement area as determined by the Z-test. The Z-test is carried out by calculating

$$Z = \frac{\bar{Y}_I - \bar{Y}_O}{0.81(\frac{1}{n_I} + \frac{1}{n_O})^{1/2}}$$

where  $\bar{Y}_I$  is the average of the natural logarithms of the inside samples and  $\bar{Y}_O$  is the average of the natural logarithms of the outside samples,  $n_I$  is the number of inside samples and  $n_O$  is the number of outside samples. The response action is considered complete if Z is less than or equal to 1.65.

Note: When no fibers are counted, the calculated detection limit for that analysis is inserted for the concentration.

2. If the abatement site does not satisfy either (1) or (2) of this Section C, the site must be recleaned and a new set of samples collected. D. Sequence for Analyzing Samples

It is possible to determine completion of the response action without analyzing all samples. Also, at any point in the process a decision may be made to terminate the analysis of existing samples

any point in the process, a decision may be made to terminate the analysis of existing samples, reclean the abatement site, and collect a new set of samples. The following sequence is outlined to minimize the number of analyses needed to reach a decision.

1. Analyze the inside samples.
2. If at least 1,199 L of air for a 25 mm filter or 2,799 L of air for a 37 mm filter is collected for each inside sample and the arithmetic mean concentration of structures per square millimeter of filter is less than or equal to 70 s/mm<sup>2</sup>, the response action is complete and no further analysis is needed.
3. If less than 1,199 L of air for a 25 mm filter or 2,799 L of air for a 37 mm filter is collected for any of the inside samples, or the arithmetic mean concentration of structures per square millimeter of filter is greater than 70 s/mm<sup>2</sup>, analyze the three blanks.
4. If the arithmetic mean concentration of structures per square millimeter on the blank filters is greater than 70 s/mm<sup>2</sup>, terminate the analysis, identify and correct the source of blank contamination, and collect a new set of samples.
5. If the arithmetic mean concentration of structures per square millimeter on the blank filters is less than or equal to 70 s/mm<sup>2</sup>, analyze the outside samples and perform the Z-test.
6. If the Z-statistic is less than or equal to 1.65, the response action is complete. If the Z-statistic is greater than 1.65, reclean the abatement site and collect a new set of samples.

[52 FR 41857, Oct. 30, 1987]





